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PYROMETRIC PRACTICE

BY

PAUL. D. FOOTE, Physicist
C. O. FAIRCHILD, Physicist
T. R. HARRISON, Associate Physicist
Bureau of Standards

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PYROMETRIC PRACTICE[a]

By Paul D. Foote, C. O. Fairchild, and T. R. Harrison

CONTENTS

	Page
I. Temperature scale	8
II. High-temperature thermometry	10
1. Introduction	10
2. High-temperature mercurial thermometers	11
3. Industrial thermometers	12
4. Emergent-stem error of mercury in glass thermometers	14
5. Other sources of error in use of high-temperature mercurial thermometers	16
6. Filling high-temperature thermometers under pressure	16
7. Improper annealing	17
8. Indicating and recording thermometers	17
(*a*) Pressure thermometers	17
(1) Liquids used in vapor-pressure thermometers	18
(2) Materials and pressures employed in liquid- and gas-filled thermometers	18
(3) Principles underlying the action of pressure thermometers	19
III. Thermoelectric pyrometry	20
1. Introduction	20
2. Metals used for thermocouples	21
3. Constancy of calibration	22
4. Reproducibility of couples	23
5. Mounted thermocouples	24
6. Indicating instruments	26
(*a*) Galvanometer method	26
(1) Resistance of indicating instruments	28
(2) Use of portable test set	33
(3) Galvanometer with variable series resistance	38
(4) Harrison-Foote method	39
(*b*) Potentiometer method	43
(*c*) Semipotentiometer method	46
(1) Northrup's method	46
(2) Northrup's method applied to continuously deflecting instruments	47

[a] Throughout this paper important technical features of different instruments are considered with a view to making available to the users of such instruments information which is essential to determining the adaptability of the several instruments to the various applications of high temperature measurement, as well as to secure the most efficient use of the instruments when installed. It is to be emphasized that no attempt is made to designate the best instrument for any given purpose, as this depends upon so many factors, such as accuracy required, cost, adaptability to working conditions, that it is necessary for the user to decide with the aid of the technical information given which particular instrument will most satisfactorily meet his needs.

III. Thermoelectric pyrometry—Continued
6. Indicating instruments—Continued Page
(*d*) Deflection potentiometer method 48
(1) Deflection potentiometer graduated to read temperature directly 53
(*e*) Temperature of the cold junctions of thermocouples 54
(1) Compensating leads 56
(2) Potentiometric compensation methods 58
(3) Compensation by use of a shunt 60
(4) Wheatstone bridge compensation 63
(*f*) Methods employed in correcting for variations in the temperature-emf relation of different couples of the same type 64
(1) Compensation by series resistance 65
(2) Compensation by shunt resistance 69
(3) Compensation by shunt and series resistance 70
(4) Summary statement on reproducibility of couples 73
(*g*) Thermocouple installations 74
(1) Special precautions 74
(2) Common return 76
(3) Wiring diagrams of thermocouple installations 78
(4) Commutating switches 82
(5) Use of junction box 83
(6) Determination of temperature of buried cold junction 87
(7) Depth of immersion of couples 88
(8) Couples purposely insufficiently immersed 89
(9) Protecting tubes for thermocouples 89
IV. General theory of optical and radiation pyrometry 94
1. Optical pyrometer temperature scale 96
2. Radiation pyrometer temperature scale 97
V. Optical pyrometry 98
1. Fery optical pyrometer 98
2. Shore pyroscope 99
(*a*) Directions for use of Shore pyroscope 99
3. Wanner pyrometer 100
4. Scimatco pyrometer 103
5. F and F pyrometer 104
6. Disappearing filament optical pyrometer 105
7. General use of optical pyrometers 108
8. Black-body and nonblack-body conditions 110
9. Temperature of glowing gauze 114
10. Use of a window 117
11. Flames and smoke 117
12. Method of sighting into a closed tube 118
VI. Radiation pyrometry 118
1. Thwing radiation pyrometer 119
2. Foster radiation pyrometer 121
3. Fery radiation pyrometer 122
4. Errors to which radiation pyrometers are subject 125
5. Advantages and disadvantages of radiation pyrometers 125
6. Black-body and nonblack-body conditions 126

Page

VII. Resistance thermometry 127
1. Three-lead Wheatstone bridge method 130
2. Four-lead Wheatstone bridge method 131
3. Four-lead potentiometric method 132
4. Deflection-galvanometer method 132
5. Summary statement on resistance thermometers 133
VIII. Recording pyrometry 134
1. Forms of temperature records 135
2. General types of thermocouple recorders for temperature-time curves 136
3. Record charts 136
4. Thermocouple recorders operating on the galvanometric principle 138
(*a*) Industrial types of recorders 141
(1) The circular-chart, single-point recorder 142
(2) Roll-chart recorders 144
5. Thermocouple recorder operating on the potentiometric principle 156
6. Transformation point indicators and recorders 161
(*a*) The Brown transformation point recorder 162
(*b*) The Leeds & Northrup transformation-point indicator. 163
IX. High temperature control 165
1. Devices used for control 169
(*a*) Automatic alarm 169
(*b*) Manual signaling 169
(*c*) Automatic signaling 170
(*d*) Automatic temperature control 174
2. Interesting examples of the utility of recorders in furnace control 181
X. Melting-point methods at high temperatures 187
1. Introduction 187
2. Use of thermoelectric pyrometers 189
(*a*) Pure materials 189
(1) General discussion 189
(2) Precision work with pure metals 192
(3) Crucibles and protecting tubes 193
(4) Table of melting points 194
(5) Construction of furnace 195
(6) General precautions 196
(*b*) Metals with impurities, and alloys 197
(*c*) Inorganic salts and silicates 202
(1) Table of melting points 206
(*d*) Technical melting-point determinations 206
(*e*) Wire method 207
3. Use of resistance thermometers 209
4. Use of optical pyrometers 210
(*a*) Metals and alloys 210
(1) Table of melting points 212
(2) Crucibles 212
(3) Furnaces 213
(4) Other methods 216
(5) Micropyrometer 216

X. Melting-point methods at high temperatures—Continued
4. Use of optical pyrometers—Continued Page
(*b*) Refractory materials ... 217
(1) Refractory oxides (with table of melting points) 217
(2) Fire bricks and ceramic materials ... 218
(3) Meaning of the melting point ... 219
(4) Conditions affecting observed values of the melting point ... 220
(5) Method of melting-point determination—sampling, grinding, and molding ... 220
(6) Time and rate of heating ... 221
(7) Type of furnace and conditions existing therein ... 221
(8) Temperature measurements ... 222
(*c*) Softening temperature of coal ash ... 223
(*d*) Table of melting points of refractory materials ... 225
XI. Standardization of pyrometers ... 225
1. Standardization of high-temperature thermometers ... 225
2 Standardization of thermocouples ... 226
(*a*) Standardization of rare-metal couples ... 227
(1) Annealing ... 227
(2) Homogeneity test ... 228
(3) Laboratory potentiometer ... 232
(4) Cold-junction ice box ... 233
(5) Calibration of primary standards ... 234
(6) Secondary or comparison calibration of rare metal couples ... 239
(*b*) Standardization of base-metal couples ... 242
(1) Use of muffle furnace ... 242
(2) Use of molten-metal bath ... 243
(3) Single-point calibration or check point ... 244
(4) Checking thermocouples in fixed installations ... 244
(*c*) Cold-junction correction ... 246
(*d*) Method of making thermocouple junctions ... 247
3. Use of standardized couples with various types of measuring instrument ... 248
(*a*) Galvanometer adjusted to read zero on open circuit, or potentiometer without cold-junction compensator ... 249
(*b*) Galvanometer adjusted to read cold junction temperature or equivalent emf on open circuit ... 249
(*c*) Potentiometer with cold-junction compensator ... 249
(1) Main scale and manually operated compensator graduated in emf ... 250
(2) Main scale and manually operated compensator graduated in temperature ... 250
(3) Main scale graduated in temperature, manually operated compensator graduated in emf ... 250
(4) Main scale graduated in emf and manually operated compensator graduated in temperature ... 251
(5) Scale graduated in emf and automatic compensator ... 251
(6) Scale graduated in temperature and automatic compensator ... 252

XI. Standardization of pyrometers—Continued Page
4. Standardization of thermoelectric instruments ... 253
(*a*) Laboratory standard potentiometer ... 253
(*b*) Adjustment of instruments for calibration ... 253
(*c*) Galvanometers ... 253
(*d*) Portable and recording potentiometers ... 255
(*e*) Potentiometer with compensator graduated in temperature ... 255
(*f*) Potentiometer with compensator graduated in emf ... 255
(*g*) Potentiometer with automatic compensator ... 255
(*h*) Instruments employing the Harrison-Foote method ... 256
5. Calibration of couple and indicator as a unit ... 257
(*a*) Galvanometric instruments ... 257
(*b*) Potentiometers ... 257
6. Standardization of optical pyrometers ... 258
(*a*) Secondary standardization ... 259
(1) Comparison source ... 259
(2) Disappearing-filament pyrometer ... 260
(3) Wanner pyrometer ... 263
(4) Fery optical and F and F pyrometer ... 266
(*b*) Primary standardization ... 267
7. Standardization of radiation pyrometers ... 273
(*a*) Primary standardization ... 274
(*b*) Secondary standardization ... 275
(1) Computation of standardization data ... 276
8. Standardization of resistance thermometers ... 278
(*a*) Ice point ... 278
(*b*) Steam point ... 278
(*c*) Sulphur-boiling point ... 278
(*d*) Computation of data ... 281
(*e*) Nickel coils ... 281
(*f*) Thermometers with indicating instruments ... 281
XII. Industrial applications of pyrometry ... 282
1. Glass manufacture ... 283
(*a*) Temperature measurements in glass melting ... 283
(*b*) General principles of annealing ... 285
(*c*) Temperature measurements in annealing ovens ... 286
2. Rotary Portland cement kilns ... 287
3. Ceramic industries ... 289
(*a*) Installations ... 291
4. By-product coke industry ... 293
(*a*) Locations for installing thermocouples ... 293
(*b*) Measurements with the optical pyrometer ... 294
(*c*) Experimental measurements in oven proper ... 295
5. Steel manufacture ... 296
(*a*) Blast furnace ... 296
(*b*) Bessemer converter ... 296
(*c*) Open-hearth furnace ... 296
(1) Dome ... 296
(2) Slag pocket ... 296
(3) Slag surface ... 296
(4) Metal bath ... 297

XII. Industrial applications of pyrometry—Continued
5. Steel manufacture—Continued Page
(*d*) Tapping and teeming 297
(*e*) Rolling, etc. 297
6. Temperatures of metal surfaces 298
XIII. Conclusion 299
XIV. Appendix 300
Index 321

I. TEMPERATURE SCALE

The ideal temperature scale is known as the thermodynamic scale. Kelvin has defined this scale to be such that "the absolute values of two temperatures are to one another in the proportion of the heat taken in to the heat rejected in a reversible thermodynamic engine working with a source and refrigerator at the higher and lower of the temperatures, respectively." The size of the unit degree is given by arbitrarily defining the temperature interval from the freezing point to the boiling point of pure water under standard conditions as equivalent to 100° C.

No thermometer or pyrometer uses this principle of temperature measurement, but scales defined by other means are supposedly in agreement with the thermodynamic scale or differ from this scale by small, known amounts.

Primarily, the high-temperature scale from 100° C to about 1500° C is defined by the expansion of nitrogen in the constant-volume gas thermometer. If an "ideal" gas existed and were employed in the gas thermometer, the temperature scale would agree throughout this range with the thermodynamic scale. Such a gas does not exist, but the corrections which are necessary to apply to the nitrogen-gas thermometer scale to make this scale agree with that of an ideal gas are fairly well known. These corrections are very small, amounting in the above case to only 0.05° at 200° C, 0.3° at 600° C, and 1° at 1200° C.

The gas thermometer is very difficult and inconvenient to operate and is not employed even for precision work except as a fundamental standard instrument. It would not be possible to calibrate all other types of pyrometer directly by comparison with the gas thermometer. Accordingly, a series of so-called fixed points have been adopted, defined by the melting or boiling points of several of the chemical elements and compounds. The temperatures at which the phenomena of melting or boiling of these materials occur have been determined up to 1500° C by the gas thermometer. In these primary experiments a thermo-

couple or resistance thermometer usually served as a transfer instrument, since, except in a few instances, it was not possible to place the bulb of the gas thermometer directly into the melting or boiling chemical element. Having obtained by means of the gas thermometer a number of fixed points which could be corrected to conform to the true thermodynamic temperature scale, it was found that the temperature scale defined by other forms of pyrometer agreed with the thermodynamic scale as closely as this scale could be determined by the gas thermometer and that the precision possible in other pyrometers is far greater than that of which the gas thermometer is capable.

Thus, it has been found that the scale defined by the platinum resistance thermometer calibrated as described later, in terms of the melting point of ice, 0° C, the boiling points of water 100° C, and of sulphur, 444.6° C, at standard atmospheric pressure, represents the true thermodynamic scale (as closely as we have any means of determining this agreement) up to 1100° C, the highest temperature at which the resistance thermometer can be operated. It has been also found that the temperature scale defined by the rare-metal thermocouple (Pt, 90 per cent Pt–10 per cent Rh) calibrated at three fixed points, such as the freezing points of zinc, antimony, and copper, agrees with the thermodynamic scale, as closely as can be determined, in the range 450 to 1100° C.

The gas thermometer has never been used with high accuracy at a temperature greater than 1550° C. Above this range the temperature scale must be defined by means of the Stefan-Boltzmann or Wien-Planck radiation laws. These laws have a theoretical significance, and experimental evidence is such that the scales defined by these two laws are in mutual agreement and that they represent the ideal thermodynamic scale.

In order to obtain as consistent a reproducible temperature scale as is at present possible, this Bureau has provisionally adopted the following procedure: In the interval −40 to 450° C the temperature scale is defined by the platinum resistance thermometer calibrated in ice, steam, and sulphur vapor (444.6° C). The scale from 450 to 1100° is defined by the fixed points copper 1083° C, antimony 630° C, and zinc 419.4° C, and interpolation between these points is based on the temperature scale defined by the rare-metal thermocouple (Pt, 90 per cent Pt–10 per cent Rh) calibrated at these three standard temperatures, using a parabolic interpolation formula. The temperature

scale [1] above 1100° C is based on the extrapolation of Wien's law, using the fiducial point m. p. gold = 1063° C, and $c_2 = 14\,350$. On the basis of the scale defined above, the following table represents the melting and boiling points of several important pure substances, the accuracy being possibly 0.1° at 500° C, 1° at 1200° C, 5° at 1500° C, and 50° at 3400° C.

TABLE 1.—Standard Fixed Points

Freezing or melting points:	°C	Freezing or melting points:	°C
Tin	231.9	Gold	1063
Lead	327.4	Copper	1083
Zinc	419.4	Nickel	1452
Antimony	630.0	Palladium	1550
Aluminum (0.997 pure)	658.7	Platinum	1755
Copper-silver eutectic	779.0	Alumina	2050
Silver	960.5	Tungsten	3400
Boiling points:			
Naphthalene	217.9°+0.058 ([a] $P-760$)		
Benzophenone	305.9 + .063 ($P-760$)		
Sulphur	444.6 + .091 ($P-760$)		

[a] P denotes pressure in mm Hg.

The above table applies for materials of the highest possible purity. The presence of impurities will, in general, lower the freezing or melting points and raise the boiling points. Methods for using the above materials for the standardization of pyrometers are discussed later.

II. HIGH-TEMPERATURE THERMOMETRY [2]

1. INTRODUCTION

High-temperature thermometry is concerned with the measurement of temperature in the range 100 to 550° C. The lower arbitrary limit corresponds to the temperature of boiling water at normal atmospheric pressure, the upper limit (550° C) is approximately the highest temperature to which a mercury-in-glass thermometer may be safely subjected.[3]

The following table gives a brief classification of the various types of high-temperature thermometers:

[1] For a more complete discussion of the temperature scale see Waidner, Mueller, and Foote, Standard Scale of Temperature, Chicago, Pyrometry Symposium.

[2] This section is a synopsis of a paper by Wilhelm, Chicago, Pyrometry Symposium.

[3] Quartz glass thermometers filled with mercury under pressure have been constructed to measure temperatures up to 750° C. Reports as to their behavior, however, have not been promising. Special glasses have been recently produced which offer some possibilities for use even above 600° C, but mercurial thermometry above 500° C is still in the development stage.

TABLE 2.—Classification of Thermometers

General classification	Subdivisions	
Mercury in glass (mercurial)	Laboratory or chemical	Etched or engraved stem inclosed scale
	Industrial.	
Indicating and recording	Pressure	Vapor pressure Liquid filled Gas filled
	Bimetallic.	
	Graphite-metal expansion.	
	Electrical	Resistance thermometer Thermocouples

2. HIGH-TEMPERATURE MERCURIAL THERMOMETERS

In the laboratory the so-called engraved or etched-stem thermometer is more generally used than the inclosed-scale (einschluss) type, probably because of the preference of American manufacturers for the former. For some classes of work, and especially under high temperature conditions, the inclosed-scale thermometer has certain advantages over the etched-scale type, in that the coloring matter in the graduation lines can not be removed by solvents, the thermometer may be repaired if only the outer tubing is broken, and parallax may be avoided by a simple procedure. The disadvantages, however, may offset the good features for the scale may become loose, and this, as well as the fact that the scale and capillary tubing may not be in close contact, may introduce uncertain errors. The computation of the correction for emergent stem is also less reliable because of the uncertainty as to the actual temperature inside the glass tubing.

FIG. 1.—*Types of high-temperature mercury-in-glass thermometers*

Fig. 1 shows characteristic types of etched-stem, high-temperature laboratory thermometers. The first instrument (*a*) is the

type used as a standard at the Bureau of Standards in the range 300 to 530° C. The second thermometer (*b*) is a continuous scale instrument used for making distillation tests and graduated from 0 to 400° C in 1° intervals, for total immersion. The third instrument (*c*) is a 20 to 750° F thermometer divided into 2° intervals for partial immersion, and is used in oil testing or other industrial work. For convenience in re-standardizing, it is desirable to have the ice-point graduations (0° C, 32° F) marked on the stem.

If it is necessary to use a thermometer for partial immersion—that is, with the bulb and only a part of the stem heated—it is advisable to use the continuous-scale type (*b*) or (*c*) rather than the standard type (*a*), especially if the enlargement in the capillary shown between the 0 and 295° marks projects out into the air, which is much cooler than the bulb, as otherwise large and uncertain errors may be introduced.

It is not advisable to graduate thermometers to be used in the range 200 to 550° C into intervals smaller than 1 or 2° C, as the maximum obtainable accuracy is from 0.1 to 0.5°C. The following table illustrates the accuracy which may be expected of high-grade thermometers used with total immersion:

TABLE 3.—Accuracy of High-Grade Thermometers

Scale range	Subdivision	Probable accuracy unstandardized	Maximum accuracy standardized	Scale range	Subdivision	Probable accuracy unstandardized	Maximum accuracy standardized
°C	°C	°C	°C	°C	°C	°C	°C
100 to 200	5	2	1	300 to 400	5	3	2
	2	1	0.5		2	1	1
	1	1	.2		1	2	.5
	0.2 or 0.5	0.5	.1	400 to 500	5	5	2
200 to 300	5	2	1		1 or 2	2	1
	2	2	.5				
	.5 or 1	1	.2				

3. INDUSTRIAL THERMOMETERS

The use of the chemical or laboratory type of mercurial thermometer is restricted to the laboratory, since unprotected glass thermometers are too frail to withstand the rough usage of the plant and shop and are comparatively difficult to read. To meet the requirement of the plant, there has been developed what is generally known as the industrial type of mercurial thermometer. As shown in Fig. 2, it is characterized by a heavy metal back and protecting tube for the bulb, large and distinct figures and gradua-

tion marks, and threaded connections for attaching the instrument readily and firmly to some part of the apparatus. This same general design of instrument is used for many different operations, covering ranges of temperature from −30 to 550° C.

It will be noted that the bulb does not come into direct contact with the substance the temperature of which is to be measured and that it is surrounded by large metal parts, which extend into a region of different temperature. These two peculiarities in the construction of an industrial thermometer must be taken into consideration in their use and calibration. Since the bulb is not in direct contact with the heated substance, the time required to take up changes in temperature is greater than for a bare bulb. The "lag" can be reduced by filling the space between the bulb and the outer wall of the casing with a good conducting medium, the most satisfactory substance for temperatures below 200° C being mercury. The lag of an instrument read in steam at 100° C with mercury surrounding the bulb has been found ½ to ⅓ as great as when powdered graphite, oil, or air was used.

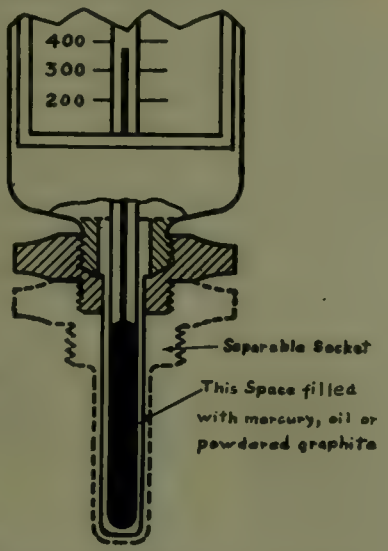


FIG. 2.—*Industrial type of mercury-in-glass thermometer*

This lag, however, may not be as great a source of error as the actual discrepancy in the readings of the thermometers when used to measure the same temperature; but, under different conditions as regards construction of the protecting case surrounding the bulb, the material into which the bulb is immersed—that is, whether liquid, vapor, or gas—the rate of flow of the material past the bulb, and the exterior conditions to which the protecting stem and casing are subjected. No data, however, are available in regard to the precise effects of these various factors, even for definite types of thermometers.

4. EMERGENT-STEM ERROR OF MERCURY-IN-GLASS THERMOMETERS

It is common practice to graduate and standardize thermometers for the condition that the bulb and the part of the stem containing the mercury are at the same temperature. This process is called graduation and standardization for total immersion. Some manufacturers, however, point part of their stock thermometers for other conditions of immersion, such as 3 inches, believing that this condition has a wider application than that of total immersion.

In the actual use of a thermometer it is quite often impossible to observe the conditions of total immersion of the mercury column, and many instruments must be used with the bulb only, or the bulb and a very small portion of the stem, heated. The remainder of the stem, containing the mercury column, is usually at a temperature considerably lower than the bulb, and the original calibration of the instrument, if for total immersion, will not hold. The thermometer reads too low under these conditions.

It is possible to compute the approximate correction to apply to the reading to reduce it to standard conditions by means of the formula [4] $S = a\ n\ (T - t)$, where S is the correction to be applied to the reading, a is a factor representing the relative expansion of mercury in glass, n is the number of degrees of the mercury emergent from the bath, T is the temperature of the bulb or bath, and t is the average temperature of the emergent mercury column. The value of a is about 0.00016 for centigrade temperatures and 0.00009 for Fahrenheit. T can be approximated by using the reading of the thermometer, and, if a higher degree of accuracy is desired, a second approximation may be made by adding the correction first found to the reading and using this value of T to obtain a second correction. The value of t can be measured roughly by an auxiliary thermometer the bulb of which is placed about three-fourths the distance down the exposed mercury column. A more precise method is to use a "faden" or thread thermometer, which is designed to give the *average* temperature of a given length of the mercury column. The theory of stem correction and the use of faden thermometers is discussed by Buckingham.[5]

In processes for which the immersion of the thermometer is definitely known thermometers may be previously graduated for

[4] Rimbach, Zeit. f. Inst., **10**, 1890, gives stem-correction tables which have been widely published. Their application is limited, however, as the data were obtained using special thermometers in distillation apparatus.

[5] B. S. Sci. Papers, No. 170: 1911.

the required immersion. Partial-immersion thermometers are subject to error unless the conditions under which the thermometer is used exactly correspond closely to those of pointing or standardization. Thus, changes in the room temperature or temperature conditions surrounding the stem may introduce errors of several degrees. However, when high accuracy is not desired, it is probably more satisfactory to use these partial-immersion instruments than to attempt a stem correction with a total-immersion thermometer. Thermometers so graduated should be marked, preferably, with a line around the stem indicating the depth of immersion and also with a statement to this effect on the stem.

Several processes in which thermometers calibrated for total immersion are used under partial-immersion conditions have been investigated at the Bureau of Standards and the following stem-correction data obtained: [6]

TABLE 4.—Stem-Correction Data for Cleveland Open-Cup Flash and Fire-Point Tester Thermometer

[Range—20 to 760° F in 2° intervals, length about 15 inches]

Thermometer reading	Degrees of mercury column emergent	Mean temperature of emergent mercury column	Stem correction
°F	°F	°F	°F
300	208	174	0.5
350	308	177	3.5
400	408	177	8.5
500	508	187	15.0
600	608	203	23.0

TABLE 5.—Stem-Correction Data for Distillation Flask Used in Distillation of Creosote Oil

[Dimensions of flask: Diameter bulb, 8 cm; diameter neck, 2 cm; height neck, 15 cm; distance bottom of outlet tube to top, 8 cm. Thermometer range, 0 to 400° C in 1° intervals; length, 40 cm]

Thermometer reading	Degrees of mercury column above bulb	Mean temperature of mercury column	Stem correction
°C	°C	°C	°C
200	208	59	4.7
250	258	111	6.0
300	308	98	10.5
350	358	95	15.5

[6] Wilhelm, B. S. Tech. Papers, No. 49.

TABLE 6.—Stem-Correction Data for Pensky-Martin Flash-Point Apparatus

Thermometer used up to 150° C, range 40 to 160° C in 1° intervals; scale length, 9.5 cm; thermometer used from 200 to 300°, range 200 to 360° in 1° intervals; scale length, 12 cm]

Thermometer reading	Degrees of mercury column emergent	Mean temperature of emergent mercury column	Stem correction
°C	°C	°C	°C
50	30	35	0.1
100	80	45	.7
150	130	55	2.0
200	10	75	.2
250	60	85	1.6
300	110	100	3.5

Empirical methods are occasionally employed for certain more or less standard operations, such as distillation, in which a total-immersion thermometer is specified, to be used without regard to the emergent-stem correction.

5. OTHER SOURCES OF ERROR IN USE OF HIGH-TEMPERATURE MERCURIAL THERMOMETERS

Aside from the error arising from the failure to observe the proper condition of immersion, there are two common sources of error which may be attributed to actual defects in the manufacture of the instrument. These are insufficient pressure above the mercury to prevent breaking of the mercury column or evaporation at the surface of the mercury and improper or insufficient annealing for use at high temperatures. This latter defect may result in a rise of the reading with continued heating amounting to as much as 30° C in extreme cases.

6. FILLING HIGH-TEMPERATURE THERMOMETERS UNDER PRESSURE

Mercury boils at 357° C at atmospheric pressure and evaporates fairly rapidly at much lower temperatures. It is accordingly desirable for thermometers used above 100° C to fill the capillary above the mercury with nitrogen under a pressure of from 2 to 20 atmospheres, depending upon the temperature range and construction of the stem. Failure to fill high-temperature thermometers under the sufficient pressure may be sometimes detected by the broken appearance of the mercury column and by drops of mercury which have distilled from the top, but often the column breaks in a part of the stem not visible and the defect is not readily noticed.

7. IMPROPER ANNEALING

Improper annealing can be detected only by repeated calibration of the instruments from time to time. Laboratories usually have facilities for checking thermometers either at the ice or steam point or for comparing them with a thermometer known to be reliable.

8. INDICATING AND RECORDING THERMOMETERS

The term "indicating" is usually employed to designate thermometers of the dial-and-pointer type, rather than those constructed of mercury and glass. Indicating thermometers may or may not be distance reading; that is, so constructed as to allow the indicator to be placed at some distance from the bulb. A distance-reading indicating thermometer is shown in Fig. 3. A recording thermometer employs a mechanism for making a continuous record of temperature on a suitable chart, as shown in Fig. 4.

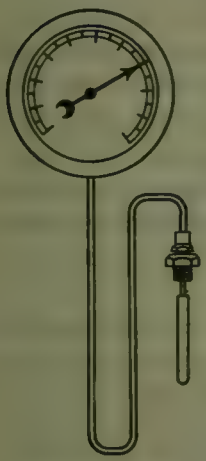


FIG. 3.—*Distance-reading indicating thermometer*

FIG. 4.—*Recording thermometer*

Indicating and recording thermometers may be divided into three general classes—electrical, pressure, and bimetallic. Electrical thermometers or pyrometers are considered later. Pressure thermometers comprise a bulb containing a liquid or gas or both connected by means of capillary tubing to some form of pressure gage. Bimetallic thermometers utilize the turning moment produced by heating a strip consisting of two metals brazed together, and having different coefficients of expansion. Graphite-metal thermometers make use of the linear differential expansion of rods of graphite and a suitable metal. The accuracy and adaptability of these types of instrument have not been sufficiently investigated to allow a more detailed discussion.

(*a*) PRESSURE THERMOMETERS.—Pressure thermometers may be subdivided into vapor-pressure, liquid-filled, and gas-filled. The thermal expansion of a liquid or gas is approximately linear with temperature; hence these thermometers have a linear tem-

perature scale. The change in vapor pressure, with temperature, however, is not linear, and the temperature scale of a vapor-pressure thermometer becomes more open at higher temperatures. Otherwise, the three types of pressure thermometers are similar in outward appearance.

(1) *Liquid Used in Vapor-Pressure Thermometers.*—The volatile liquid used in a vapor-pressure thermometer must possess certain well-defined characteristics. It should be stable, readily obtained, and purified, should not act upon the metals with which it is in contact, must have a sufficiently high pressure at the lower temperatures to insure a readable temperature scale, and its critical temperature must be higher than the highest temperature to be measured. The vapor pressures of the liquids available have been determined over wide ranges, and it is usually possible by making use of such data to select the most suitable liquid for a given range of temperature or, if a liquid be given, to predict the temperature range within which it can be used and the pressures which will be developed. In general, the temperature and pressure range for a given liquid will be included between its boiling point at a pressure of one atmosphere and its critical temperature. Although these limits are very arbitrary, they are convenient for reference.

The following table gives the boiling points at a pressure of 1 atmosphere and the critical temperatures and pressures for various liquids suggested for use in vapor-pressure thermometers.

TABLE 7.—Boiling Points and Critical Data of Liquids Suggested for Use in Vapor-Pressure Thermometers

Substance	Boiling point	Critical temperature	Critical pressure
	°C	°C	Atmospheres
Ethyl alcohol	80	243	63
Benzine	80	288	48
Water	100	365	195
Toluene	111	320	
Aniline	184	426	52

(2) *Materials and Pressures Employed in Liquid and Gas-filled Thermometers.*—Pure materials chemically inactive upon the bulb are requisite, and the highest temperature measured should not exceed the critical temperature of the liquid. Alcohol, which has been used in liquid-filled thermometers, is not satisfactory above 200° C. Aniline has been suggested for temperatures below

400° C. Mercury, however, is used almost exclusively for temperatures up to 500 or 600° C. Gas-filled thermometers using preferably nitrogen are not satisfactory much above 500° C, since the metal bulbs become permeable to the gas at high temperatures. The initial pressure and the pressure range of a liquid or gas filled thermometer varies according to the pressure range of the gage and the temperature range.

(3) *Principles Underlying the Action of Pressure Thermometers.*—The action of the vapor-pressure thermometer depends upon the fact that the pressure inside the thermometer is determined solely by the temperature of a free surface of the liquid. It follows, therefore, that the thermometer must be so constructed that one free surface is always in the bulb. If this condition is fulfilled the reading of the instrument is not sensibly affected by changes in the temperature of the gage and capillary. This is a decided advantage over other types, when the connecting tubing and gage are subjected to considerable changes in temperature. If the vapor-pressure thermometer is not filled properly—that is, the proportion of liquid is too great or too small as compared with the volume of the bulb, capillary, and gage—very large and uncertain errors may be introduced. As an example, consider the condition where the bulb is much hotter than the capillary. The liquid is condensed in the capillary, and there should be sufficient liquid to entirely fill the capillary and partly fill the bulb. If the liquid fills the gage and only partly fills the capillary, and there is no liquid in the bulb, the capillary will contain the free surface of the liquid and the temperature indicated will be that corresponding to this portion of the capillary as shown in Fig. 5. This temperature, which may be several hundred degrees lower than that of the bulb, will be indicated instead of the true temperature of the bulb.

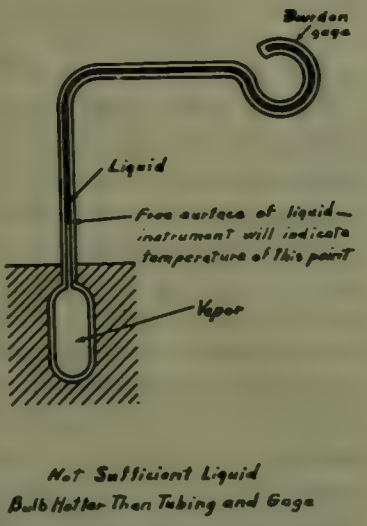


FIG. 5.—*Vapor-pressure thermometer with insufficient liquid*

Gas and liquid filled thermometers operate on the principle of thermal expansion. They are entirely filled with either the liquid or the gas. The expansion of the liquid or gas in the bulb is transmitted through capillary tubing to the pressure gage. These instruments are subject to error if the gage and capillary are at a different temperature from that at which they were calibrated. This error may be made negligible in many instances by reducing the volume of the capillary and gage as compared with that of the bulb or by using a compensator in the lead. Such a compensator, however, will not eliminate errors due to the heating or cooling of the capillary alone.

III. THERMOELECTRIC PYROMETRY

1. INTRODUCTION

Seebeck discovered in 1821 that if in a closed circuit of two metals the two junctions of the metals are at different temperatures an electric current will flow in the circuit. For example, if the ends of an iron and of a copper wire are fused together and one of the junctions is heated (within certain temperature limits), positive current will flow in the direction copper to iron at the hot junction or iron to copper at the cold junction. Two causes contribute to the production of the emf which actuates the current. An emf exists between two different metals placed in contact the magnitude of which depends upon the temperature and upon the metals used. Also, if a wire of homogeneous material is heated at one end, an emf is developed between the hot and cold ends of the wire the magnitude of which depends upon the metal and upon the differences in temperature of the ends. These two emfs are known as the Peltier emf and the Thomson emf, respectively. The total emf acting in the copper-iron circuit described above is the sum of the Peltier emf at each junction and the Thomson emf over each wire, consideration being given, of course, to the algebraic signs of these four emfs. The total emf acting in a circuit of two dissimilar metals thus depends upon the temperatures of the two junctions. If the temperature of one junction is fixed at that of the room or of melting ice, etc., the temperature of the other junction can be determined by measuring the emf developed in the circuit. This is the basic principle of thermoelectric pyrometry. In general, the thermoelectric circuit consists of more than two different metals, copper and manganin being introduced by the instrument employed for measuring the emf. However, it

may be shown that the insertion of a different metal in the circuit produces no effect upon the total emf if both ends of the introduced metal are at the same temperature. The emfs developed by thermocouples are small, usually a few thousandths of a volt. To measure such small emfs, special types of sensitive voltmeters (millivoltmeters) or indicators are required. For any particular type of couple these instruments may be graduated to read temperature directly instead of emf.

A simple thermoelectric pyrometer consists of three parts: (*a*) The thermocouple of two different metals or alloys having a fused junction (the hot junction), which is inserted into the furnace, and the cold junctions, which protrude from the furnace and are maintained at some fixed temperature, such as that of the room or of melting ice; (*b*) two lead wires, usually of copper running from the cold junctions of the thermocouple to the indicator; (*c*) the indicator, which may be a millivoltmeter, a potentiometer, or a special type of instrument embodying both of these principles, and may be graduated to read either emf, temperature, or both.

2. METALS USED FOR THERMOCOUPLES

Although any two dissimilar metals might be employed for a thermocouple, certain combinations are unsatisfactory because of the very small emfs which can be developed and because of the fact that with some combinations the emf may first increase, then decrease, become zero, and change sign as the temperature increases. Obviously, desirable properties for a thermocouple are:

1. Capability for resisting corrosion and oxidation.
2. The development of relatively large emfs.
3. A temperature-emf relation such that the emf increases continuously with increasing temperature over the temperature range employed.

For general work at higher temperatures several different types of couples are employed in this country. Up to 360° C for extreme precision or to 500° C for a precision of 5 or 10° C the couple may consist of one wire of copper and the other wire of constantan. Iron-constantan or nichrome-constantan may be employed for technical processes below 900° C. For operation below 1100° C special patented alloys of chromium and nickel and of aluminum and nickel, "chromel-alumel" or nichrome-alumel are very satisfactory even for continuous service. For the temperature range 300 to 1500° C the Le Chatelier couple should be employed. This

couple consists of one wire of platinum and the other wire an alloy containing 90 per cent platinum and 10 per cent rhodium. Other alloys and metals may be employed for special work, but the above combinations are sufficient for almost all technical processes carried on in the temperature range 0 to 1500° C. No satisfactory couple has been developed for operation above 1500° C. There are several metals and numerous alloys which melt at much higher temperatures, but all so far known are subject to serious disadvantages which make their application useless commercially. For example, iridium, 90 per cent iridium–10 per cent ruthenium, may be used up to 2000° C, but the couple is so costly as to be prohibitive, is so fragile and brittle that a slight jar will fracture it, and the iridium rapidly volatilizes even at 1200° C. Tungsten-molybdenum could be employed possibly up to 2400° C, but both of these metals oxidize so readily that extreme care must be taken by use of hydrogen to prevent oxidation. A satisfactory method for thus protecting such a couple for technical purposes has never been developed. The peculiar fact that nickel is readily oxidized by heating in air and soon becomes so brittle as to be useless, but when alloyed with chromium or aluminum the material resists oxidation and does not crystallize so rapidly, suggests the possibility that certain alloys of tungsten may be developed which would not exhibit the property of rapid oxidation characteristic of the pure metal. Such alloys might prove of great value in thermoelectric pyrometry.

3. CONSTANCY OF CALIBRATION

A further essential property of a thermocouple is constancy of calibration. Alterations in the calibration may be due to various causes. Inhomogeneity may develop on account of contamination by fumes from the furnace, metallic vapors, by oxidation, etc. Contamination may be usually prevented by the use of suitable protecting tubes surrounding the couple, and the effect of contamination may be minimized by using wire of large cross section. Different furnace conditions and different types of couples require different methods of protection against contamination. Each process must be considered individually. For example, a platinum couple is usually protected against contamination by means of refractory porcelain tubes; but, if the atmosphere surrounding the platinum is reducing, the use of porcelain may do more harm than good. In this case the reducing atmosphere changes the silica of the porcelain into silicon, which readily attacks the plati-

num. The emf of some couples gradually decreases with use. The (platinum, platinum 90 per cent-iridium 10 per cent) couple has not proved very satisfactory for this reason, although it develops a much larger emf than the platinum-rhodium couple. The iridium gradually distills from the alloy wire, especially above 1200° C, requiring frequent recalibration of the couple.

4. REPRODUCIBILITY OF COUPLES

When thermocouples are employed in the laboratory for scientific purposes, it is desirable, although not of serious importance, that the calibration or temperature-emf relation of couples of the same type be exactly similar. However, in the industrial plant this question of reproducibility is of considerable moment. The indicating instruments are usually graduated in degrees of temperature, and the graduation applies for one definite temperature-emf relation only. If the temperature-emf relations of various couples of the same type are not similar, corrections must be applied to the readings of the indicator, and these corrections will be different for each couple. When several couples are operated with one indicator, and when the process is such as to require a frequent renewal of couples, the applying of these corrections becomes very troublesome. For extreme precision it is always necessary to apply such corrections, but for most industrial processes thermocouples which are sufficiently reproducible can be secured, so that the corrections are negligibly small and may be omitted. Thus the temperature-emf relations of different homogeneous and uncontaminated chromel-alumel or iron-constantan couples should not vary in general by more than 10 or 15° C and of platinum, platinum-rhodium couples by more than 2 or 3° C. In fact, specifications for chromel-alumel and iron-constantan couples may be written as closely as ±15° F and in certain instances specifications to ±5° F are being satisfied. There are no industrial processes carried out under such accurate temperature control that the variations in the calibration of different new platinum, platinum-rhodium thermocouples of the same type warrant consideration. The variations in the calibration of different base-metal couples are frequently corrected by use of series or shunt resistance, but most methods so far devised are rather unsatisfactory, and some of the compensation methods employed may develop after continued use larger errors than those arising from the irreproducibility of the couple, as will be shown later. The

above statements in regard to reproducibility of different couples of the same type apply only for new couples. After a couple has been used for some time, especially a base-metal couple, or has become contaminated in any manner, the calibration may change considerably. All thermocouples should be checked frequently as installed to determine whether the calibration has altered.

5. MOUNTED THERMOCOUPLES

The question of properly mounting and protecting a thermocouple is of great importance. The type of protection necessary depends upon the particular industrial process for which a couple is employed and will be considered in some detail later. In the laboratory if it is desired to measure the temperature of a perfectly clean porcelain platinum-wound furnace containing no material such as iron, etc., which could contaminate platinum, a rare-metal couple may be used without any protection whatever. For most laboratory experiments, however, a rare-metal couple requires protection. The usual rare-metal couple consists of wires 0.5 mm or preferably 0.6 mm in diameter and from 50 to 125 cm in length. Wires as small as 0.1 mm, and even less, are frequently used for special research work. One or both of the wires are insulated by threading them through small porcelain or quartz tubes. In measuring temperatures below about 1400° C, two-hole porcelain tubes are very useful for insulating both wires, but for higher temperatures separate tubes should be used. The hot junction of the couple is made by fusing the two wires in an arc or oxygen-illuminating gas flame. The couple and insulating tube are inserted in a small outside protecting tube of porcelain, glazed on the outside only, or of fused silica, hemispherically closed at one end. On the open end of the protecting tube may be mounted the head of the couple, which serves as a handle and as a support for rigidly holding the wires of the couple. The couple wires frequently extend beyond the head of the couple, so that their ends may be maintained at some controlled cold-junction temperature. Usually the wires terminate in binding posts on the couple head, in which case the cold-junction temperature may be controlled by water jackets, or may be allowed to remain that of the surroundings, or the couple may be fitted with one of the various devices discussed later for the elimination of cold-junction errors.

Base-metal couples for laboratory use may be constructed in much the same manner described above and may be made of wire

as small as No. 20 or of very much smaller wire for certain types of research at lower temperatures. Small wires, however, are readily oxidized completely at high temperatures, so that for con-

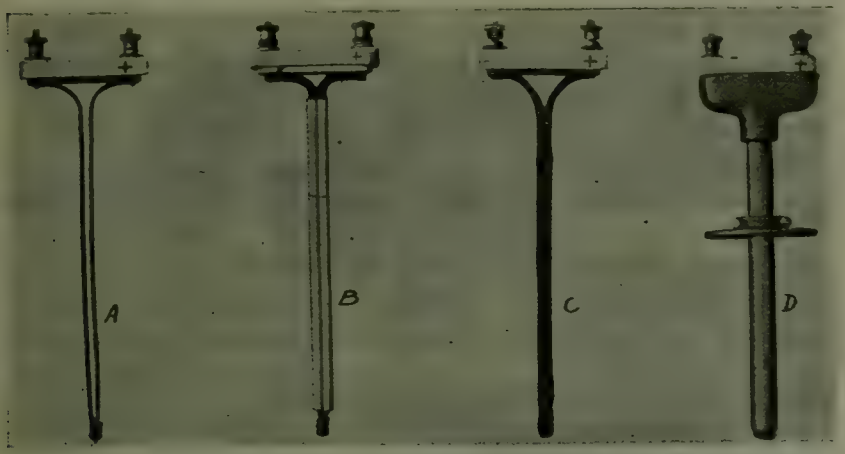


FIG. 6.—*Types of commercial thermocouples*

A, bare couple; B, couple with porcelain insulators; C, couple with asbestos insulation; D, ordinary mounting of base-metal couple in an iron protecting tube

tinuous operation in industrial installations the couples are constructed of No. 8 or No. 6 wire, or of still larger wire when there is danger of contamination. The hot junction is fused as in the case of rare-metal couples, and usually the two wires are twisted

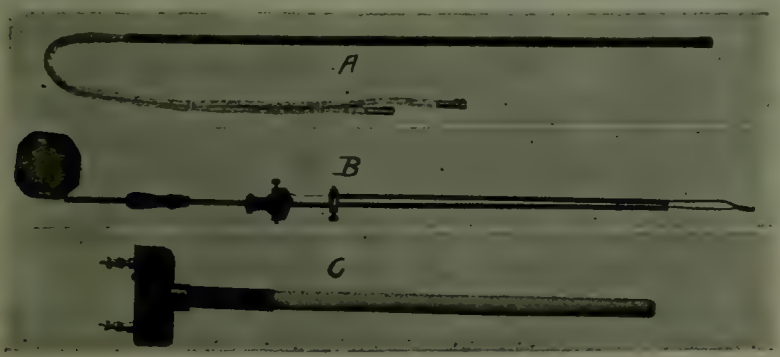


FIG. 7.—*Types of commercial thermocouples*

A, Iron-constantan couple in iron protecting tube for use below 900° C. B, extensible chromel-alumel couple for molten brass. The exposed junction which is immersed in the molten brass without protection is renewed from the magazine of reserve wire. C, porcelain or fused silica protecting tube and mounting for rare-metal couples

for a few turns at the hot junction in order to give greater mechanical strength to the joint. The two wires are insulated by fire-clay insulating tubes or by asbestos sleeving or cord, and are

connected to a suitable couple head, forming the cold junction. For severe use it is necessary to incase the couple in a protecting tube of steel, chromel, porcelain, fire clay, etc. A different form of base-metal couple consists of an outer tube of iron and an inner wire or rod of constantan. The two are fused at one end into a neat joint, forming the hot junction, and are insulated from each other up to the couple head or cold junction. The couple is thus mechanically stronger than couples formed of two wires, and, when used without an additional protecting tube, is somewhat less liable to contamination than the bare two-wire couple of the same type, since the constantan, which is probably more susceptible to contamination than the iron, is protected by the iron tube. However, couples of this type must be further protected by outer tubes, if subjected to severe furnace conditions. Figs. 6 to 10 illustrate several couples and mountings of different types. Couples of various shapes may be constructed for special purposes. Thus, frequently it is desirable to make a right-angle bend in a couple. Usually a mounted couple will stand bending into various shapes; but, before bending, one should examine the couple and temporarily remove porcelain tubes or insulators liable to be broken by the process.

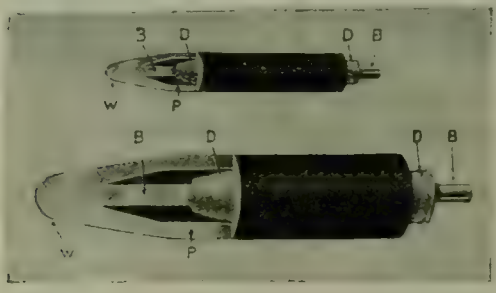


FIG. 8.—*Types of commercial thermocouples*

End section showing the outer tube P, which is one element, welded to the other element B at W. The inner rod is insulated from the tube by asbestos cord

6. INDICATING INSTRUMENTS

The indicating instruments which are connected to the thermocouple are of three general types—those operating upon the galvanometric principle, as an ordinary voltmeter, those operating upon the potentiometric principle, and those operating upon a combination of these two principles. The first two types of instrument have been made automatically recording, as will be discussed elsewhere.

(*a*) GALVANOMETER METHOD.—Galvanometers for measuring the emf developed by a thermocouple usually operate on the d'Arsonval principle, having a moving coil mounted between the poles of a permanent magnet. The deflection of the pointer

or boom attached to the coil is proportional to the thermoelectric current passing through the coil, and the current is proportional to the emf developed by the couple. Different methods for mounting the coil are employed. The coil may be suspended both above and below by phosphor-bronze suspensions, and,

FIG. 9.—*Types of commercial thermocouples*

Interior of thermocouple head showing the cold-junction compensator described later under Fig. 37

although many foreign instruments of this type have proved very delicate, in one of the latest forms the American instrument, using the double-suspension galvanometer, may be subjected to severe handling without any injury whatever. The mounting

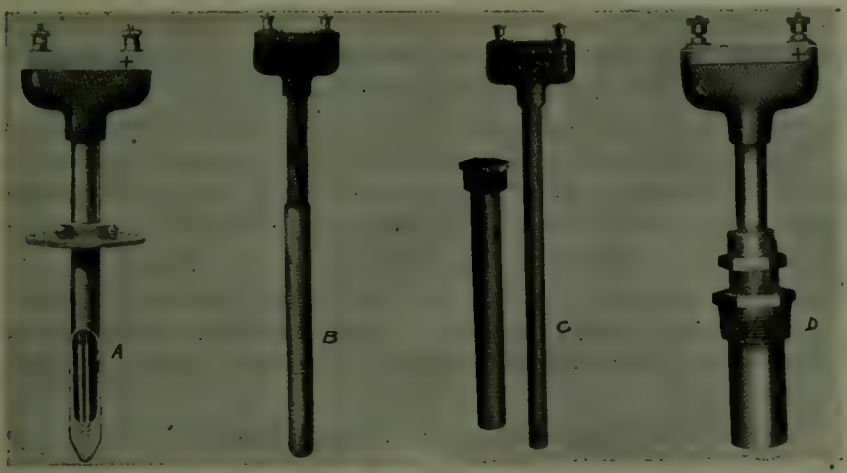


FIG. 10.—*Types of commercial thermocouples showing special mountings for base-metal couples*

A, cross section of the end of a thermocouple showing the hot junction welded to the end of the protecting tube to reduce thermal lag. A couple of this type is useful in the measurement of rapidly changing temperatures. B, couple protected by nichrome tube. C, couple with extra protecting sheath. D, couple for pipes and mains. The protecting tube is threaded into the main

consisting of an upper suspension and a lower pivot has been used extensively.

A unipivot system is employed by one English and one American manufacturer. The pivot is at the center of the coil, and the movement is balanced so that the center of gravity of the whole

moving system is at the point of the pivot. In all pivot instruments the pivots must fit in well-made jewel bearings in order to reduce friction to a minimum. The advantages claimed for the unipivot system are (1) the axis of rotation of the coil passes through the pivot point thereby minimizing undesirable effects of friction; (2) the pivot and jewel may be separated during clamping; and (3) a heavier coil may be employed.

The scale of the instrument may be graduated to read emf or temperature. By use of a series resistance mounted inside the galvanometer case and an extra binding-post terminal, two scale ranges may be obtained, one for base-metal couples of a certain type and the other for the rare-metal Le Chatelier couple. For precision work the scale should have a mirror placed parallel to the scale and extending throughout its length for use in eliminating the error in reading due to parallax. While obtaining a reading the observer should use one eye only, and he should move his head to the position where the pointer and its reflected image in the mirror coincide.

Indicators may be obtained in either the switchboard type or portable type. The former is desirable for permanent installations. Many switchboard instruments have a scale ruled on ground glass and illuminated from the rear by an electric lamp. The black enameled graduations upon the scale thus appear distinct against a bright background. Paper scales illuminated from the front are frequently employed. Usually high precision is not required of an instrument of the switchboard type, so that the graduations can be coarse and readable at some distance. However, it is possible to obtain highly accurate switchboard indicators, in which case the scale should have a mirror to permit readings without parallax.

If the instrument is mounted in a dusty room, it should be protected by an outside case of steel or wood having a glass front, as the presence of dirt filtering into the interior of the galvanometer may seriously affect its calibration. Both the wall type and the portable indicator, especially the latter, must be sufficiently robust to withstand reasonably severe usage.

(1) *Resistance of Indicating Instruments.*—When operated at the highest safe-working temperatures, most base-metal couples develop a maximum emf of less than 70 millivolts, and the Le Chatelier couple develops an emf of about 16 millivolts. In order to measure such small emfs accurately, a very sensitive indicator

or millivoltmeter is required. On the other hand, the instrument must be robust and able to withstand rough handling, to which it is necessarily more or less subjected. These conditions of mechanical robustness and of high accuracy as a pyrometer indicator are difficult to satisfy. The deflection of the pointer of the millivoltmeter is approximately proportional to the current flowing through the coil, so that, if the resistance of the total thermoelectric circuit is very low, relatively large currents are obtained, with a resulting high torque or turning moment on the movable coil. Thus when the current is high, the construction of the indicator may be made very robust. Relatively strong springs, against which the turning moment of the coil carrying the current is balanced, may be employed and less attention need be given to the friction of the pivots in their bearings. The torque may be made high without greatly increasing the resistance of the circuit by using a large number of turns of copper wire in the coil Copper possesses a large temperature coefficient of resistance, so that ordinarily the calibration of an instrument having its entire electrical circuit of copper would be affected by the temperature. However, by use of shunt and series resistances of a certain type it is possible to reduce these errors due to temperature changes to an amount of negligible importance. Other satisfactory methods for reducing this error are also employed. Thus it is not difficult to construct a sufficiently sensitive millivoltmeter of low resistance. The objection, however, from the pyrometric standpoint, to such an instrument used as a simple galvanometer has led to the development of indicators of considerable resistance, as will appear immediately. These are made by placing a high resistance of zero temperature coefficient, such as manganin, in series with the coil and, by increasing the number of turns on the moving coil, to compensate for the decrease in sensitivity arising from the increased resistance. The so-called swamping resistance of zero temperature coefficient may be so proportioned relative to the copper that, due account being taken of the temperature coefficient of elasticity of the springs, the instrument as a whole possesses a negligible temperature coefficient. The use of high resistance greatly diminishes the magnitude of the current flowing through the coil, and thus decreases the torque-producing deflection. Hence, attention must be given to the elimination of bearing friction as far as possible. Thus the instrument is necessarily more delicate than an indicator of low resistance and of the same type. The ad-

vantage of an instrument having a high resistance is demonstrated by the following discussion.

When a thermocouple in a furnace is connected to a millivoltmeter, the emf of the couple causes a current to flow around the circuit, which in accordance with Ohm's law is equal to $\frac{e}{R}$ where e is the emf developed by the couple and R is the total resistance of the circuit. Thus, if the temperature of the furnace remains constant, e is constant, but the deflection of the instrument will be altered by changes in the resistance R. Hence, any variation in R which produces a change in the reading of the instrument would be interpreted as a change in the temperature of the furnace, although actually this is constant. The total resistance of the circuit may be divided into three parts—R_g the resistance of the millivoltmeter, R_L the resistance of the line, and R_c the resistance of the couple. If these resistances are properly proportioned, the effect upon the reading of the indicator of any changes likely to occur in the resistance R can be reduced to practically zero. This condition is realized when R_g, the resistance of the galvanometer, is sufficiently high compared with $R_c + R_L$, the resistance of the external circuit. Suppose that the indicator has an emf scale graduated to read the potential difference at its terminals. The relation between the reading of the instrument e_o and the true emf, e, of the couple to which it is connected is given by the following equation:

$$e_o = \frac{R_g}{R_g + R_c + R_L} e.$$

Thus, if R_g is very large compared to $R_c + R_L$, the ratio $\frac{R_g}{R_g + R_c + R_L}$ is practically 1, and the reading of the galvanometer gives the true emf of the couple.

Robust indicators are now obtainable having a resistance of 300 to 1200 ohms. As a particular example of the behavior of such an instrument, values of the factors occurring in the above equation will be considered for an actual thermocouple installation.

Galvanometer resistance $= R_g = 300$ ohms.
Couple resistance $= R_c = 1$ ohm.
Line resistance $= R_L = 1$ ohm.

Whence,

$$e_o = \frac{R_g}{R_g + R_c + R_L} e = \frac{300}{300 + 1 + 1} e = 0.993e.$$

Thus, if the galvanometer is graduated to read potential differences at its terminals, the reading of the instrument gives the true emf of the couple to within 0.7 per cent. Instruments having a resistance as low as 10 ohms, or even less, are in extensive use. Suppose a galvanometer 10 ohms in resistance, calibrated to read potential differences at its terminals, is used in the circuit described above.

$$e_0 = \frac{R_g}{R_g + R_c + R_L} e = \frac{10}{10 + 1 + 1} e = 0.83e.$$

Thus, if the galvanometer is graduated to read potential differences at its terminals, the emfs indicated by the instrument would be 17 per cent less than the true emf of the couple. Such large errors are compensated for by arbitrarily graduating the scale to read the emf of the couple when the resistance of the couple and line has a certain fixed value; in the above example, 2 ohms. The instrument will accordingly read correctly for this external resistance, but will read in error if the external resistance is altered. Bad contacts, deterioration of the couple from oxidation, change in depth of immersion, temperature coefficient of the copper lead wires, etc., may at any time alter the resistance of the external circuit. Let us compare the behavior of the 300-ohm instrument and the 10-ohm instrument, assuming both instruments are compensatingly graduated to read correctly for an external resistance of 2 ohms, when for one of the several reasons cited above the external resistance varies slightly. The relation between the potential drop e_0 across the terminals of the galvanometer and the emf, e, of the couple is as follows, where the total line resistance $R' = R_c + R_L$:

$$e_0 = \frac{R_g}{R_g + R'} e.$$

Hence, e_0 is always less than e. Suppose, however, for a certain line resistance, $R' = R'_0$, the scale of the indicator is arbitrarily graduated so that the reading e' on the galvanometer equals the true emf, e, of the couple. The relation between the scale reading and the potential drop across the terminals of the instrument must be accordingly:

$$e' = \frac{R_g + R'_0}{R_g} e_0 = F e_0,$$

where F is a constant. The graduations on the scale are such that for any deflection of the pointer the scale reading is a con-

stant F times the potential drop across the galvanometer terminals. On substituting this value of e_o in the above equation we obtain

$$e' = Fe_o = F\frac{R_g}{R_g + R'}\, e.$$

The following table illustrates the per cent error in the indicated emf, e', computed from the above equation, when the line resistance R' has the values 1, 2, 3, and 4 ohms, respectively, when R_g, the resistances of the indicators are 300 and 10 ohms, respectively, and R'_o, the *normal* line resistance, is 2 ohms.

FIG. 11.—*Types of indicating galvanometers*

In the instrument illustrated the moving element is supported by a phosphor-bronze filament and by one pivot. Most of the weight of the moving coil is carried by the suspension. The instrument has a very high resistance and a very long scale, the pointer swinging through an arc of nearly 180°. These indicators are equipped to use the device illustrated by Fig. 37 for cold-junction compensation

TABLE 8.—Error Due to Change in Line Resistance

Line resistance (ohms)	Error in indicator reading		Error in degrees at 1000° C	
	300 ohms	10 ohms	300 ohms	10 ohms
	Per cent	Per cent	°C	°C
1	+0.33	+ 9.1	+3.3	+ 91.0
2	±0.00	± 0.0	±0.0	± 0.0
3	− .33	− 7.7	−3.3	− 77.0
4	− .66	−14.3	−6.6	−143.0

Thus when both instruments read correctly for an external resistance of 2 ohms, if the external resistance is increased by an ohm, the indicator of low resistance is in error by 7.7 per cent, or about 77° C at 1000° C, while the instrument of high resistance still reads practically correct. Considerations such as the above emphasize the importance of using a galvanometer having a resistance of 300 ohms or more. In actual operation the line resistance may change by several ohms on account of bad contacts and deterioration of the thermocouple.

FIG. 12.—*Types of indicating galvanometers*

These portable indicators are made in two types, one having a resistance of about 5 ohms per millivolt and the other about 25 ohms per millivolt

It must not be inferred from the above discussion that *all* indicators of high resistance are necessarily superior to *all* indicators of low resistance Excellent workmanship and mechanical details, robustness, etc., frequently lead to the choice of certain instruments having a resistance less than 100 ohms, especially in the case of recording millivoltmeters; but, nevertheless, the consideration of the resistance of the instrument is a matter of extreme importance, and a simple galvanometric indicator of low resistance is always subject to the large errors arising from small changes in the resistance of the circuit. This statement does not apply to the compensated galvanometer, or to semipotentiometric instruments described later. Figs. 11 to 19 represent typical indicating galvanometers of American manufacture.

(2) *Use of Portable Test Set.*—On account of the errors which may be introduced in the reading of a galvanometer when the resistance of the line or couple varies, it is important to have some means for measuring this resistance occasionally. This is, of course, especially necessary when the resistance of the galvanometer is low. Every plant requiring a large thermocouple installation with simple galvanometric indicators should have a

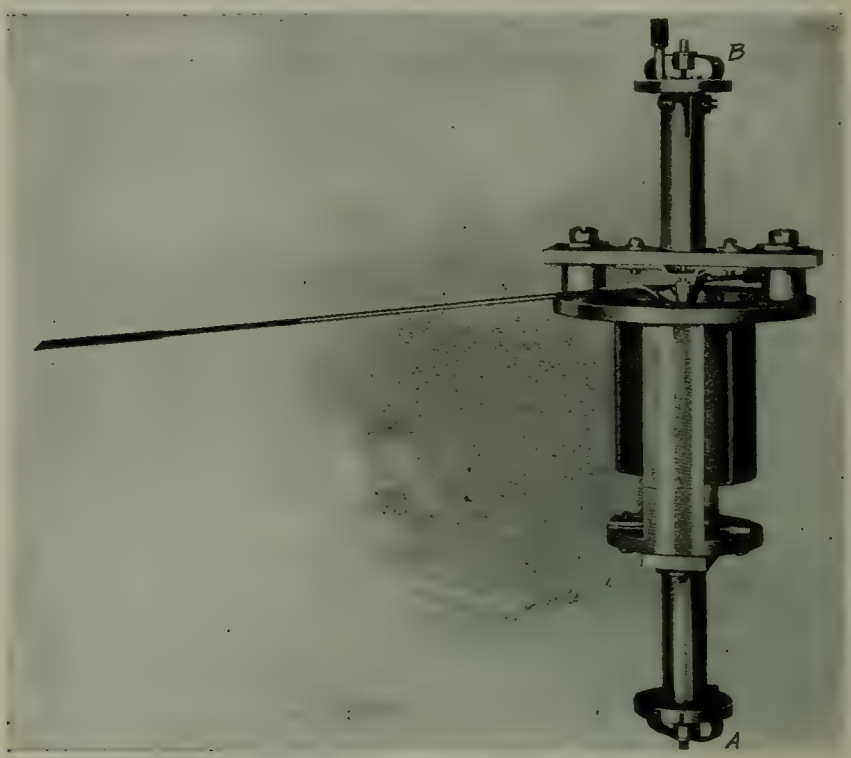


FIG. 13.—*Types of indicating galvanometers*

Moving element of a double suspension instrument in which the suspensions are kept under tension by the springs A and B. The tension is sufficient to maintain axial alignment of the coil without precise leveling. The instrument has a high resistance, about 50 ohms per millivolt

FIG. 14.—*Types of indicating galvanometers*

The wall-type instruments shown can be obtained with resistances as high as 550 ohms for base-metal couples and 200 ohms for rare-metal couples

portable Wheatstone bridge or "test" set for this purpose. Fig. 20 illustrates a simple and inexpensive instrument. Operating directions are mounted in the cover of the instrument for con-

FIG. 15.—*Single-pivot movement used in indicators shown in Fig. 14*

The pivot is at the center of gravity of the moving system

venient reference. Disconnect the pyrometer indicator from the circuit and connect the two line wires to the X terminals of the

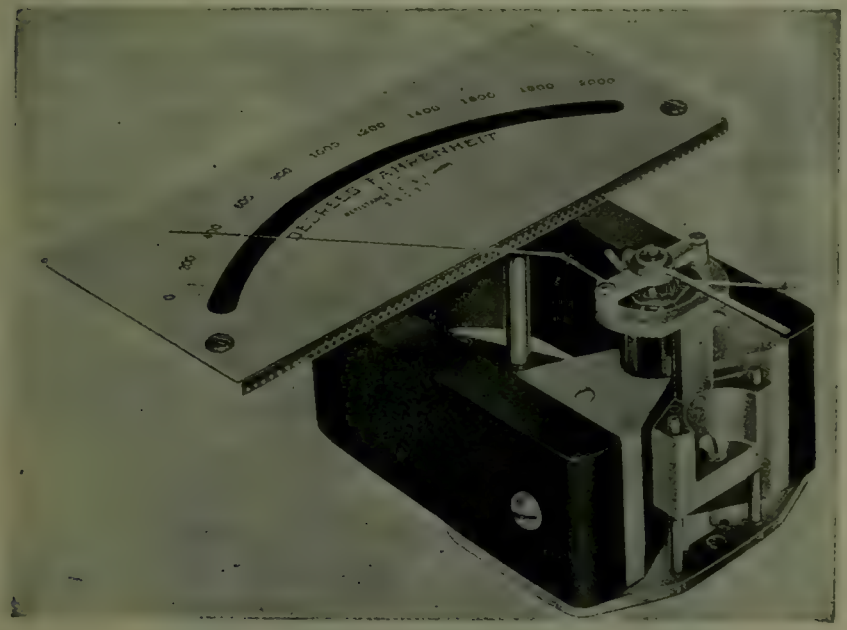


FIG. 16.—*Types of indicating galvanometers*

In this instrument the moving element is shown partially removed from the magnet. These indicators have resistances approximately 15 to 30 ohms per millivolt

test set. Note the measured resistance, reverse the + and − lead wires at X and obtain a new setting. The mean of the two observations is the resistance of the circuit. The two settings

will be different because of the emf developed by the couple. In measuring the resistance of the line care must be taken, if two indicators or an indicator and recorder are operated in parallel

FIG. 17.—*Types of indicating galvanometers*

A wall-type indicator which is made in two general types, "high resistance" and "low resistance." The former has a resistance of from 150 to 300 ohms and the latter about 8 ohms, for use with chromel-alumel couples

on the same couple, to see that both instruments are disconnected from the circuit during the measurement of the resistance; otherwise the instrument left connected acts as a shunt upon the line. If the resistance of the line and couple is found to be much higher than that for which the indicator was designed, short-circuit the line at the cold junction and determine whether the fault is in the couple or the line. If in the former, it usually means that the couple is broken or requires replacement. By making occasional

FIG. 18.—*Types of indicating galvanometers*

A wall-type indicator made with a resistance of approximately 10 ohms per millivolt

observations of this kind serious faults may be detected long before they would be suspected from the resulting low values in the indicated temperatures.

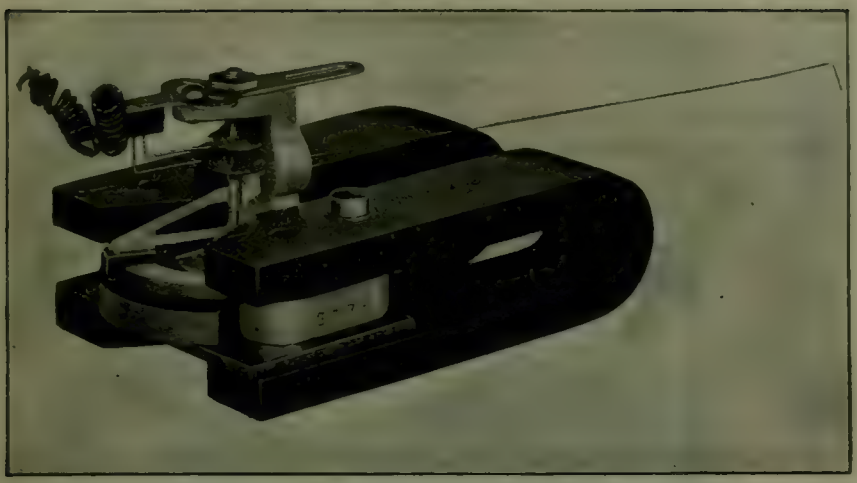


FIG. 19.—*Magnet and moving element of an indicating galvanometer which is made with a resistance of approximately 20 ohms per millivolt*

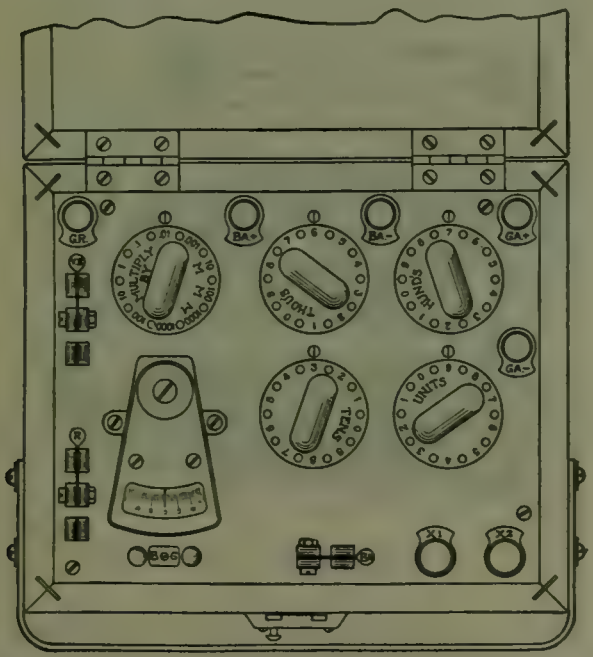


FIG. 20.—*"Test set" or Wheatstone bridge for measuring line resistance*

(3) *Galvanometer with Variable Series Resistance.*—As shown above, galvanometers, especially those of low resistance, are usually calibrated to read correctly for a definite line resistance. Suppose an indicator is desired for operation on a line of which the resistance changes from practically zero to 10 ohms. The instrument is calibrated to read correctly for a line resistance of

FIG. 21.—*Type of indicating galvanometer embodying Harrison-Foote method*

10 ohms. Located in the case of the galvanometer and in series with the line is a variable resistance from zero to 10 ohms. As the resistance of the line increases the variable resistance is decreased by manual adjustment, so that the sum of the line resistance and the variable series resistance always equals 10 ohms. The dial of the variable resistance is graduated to read the amount

of resistance cut out of the circuit. Hence it should be set at the resistance of the line and couple. This value may be determined by means of a test set as discussed above. Thus the method is of great value for precision work with a galvanometric indicator. The principal objection to it, which applies to all galvanometric indicators so far described when used for accurate measurements, is the necessity of measuring the resistance of the line and couple, this latter measurement requiring the use of a test set or similar device.

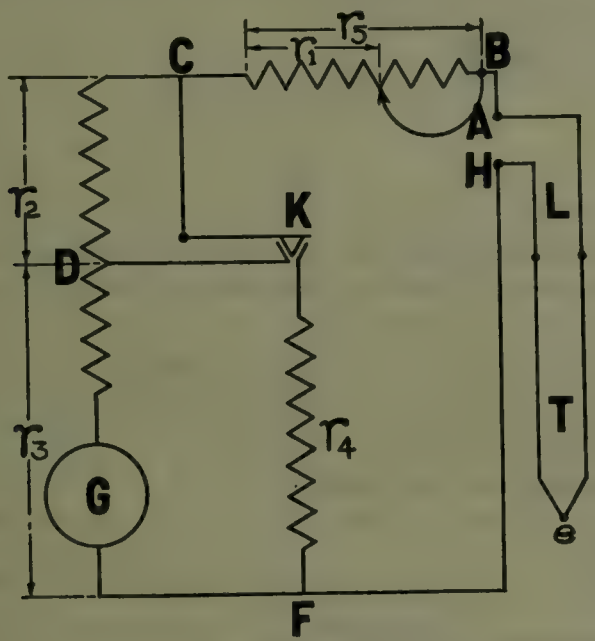


FIG. 21a.—*Wiring diagram for Fig. 21*

(4) *Harrison-Foote Method.*[1]—In this method the necessity of using a separate instrument to measure the line resistance has been eliminated. The method is employed in the Brown Improved Heatmeter shown in Fig. 21. The circuit *CDGF*, Fig. 21a, is an ordinary millivoltmeter in which *G* represents the moving coil. In series with this is an adjustable rheostat *CB*. The maximum value r_5 of this resistance is chosen equal to the maximum value of the resistance of the line and couple likely to occur in practice. A convenient value is 15 ohms. With the slide of the rheostat set for the maximum resistance, $r_1 = r_5$, the instrument is calibrated in terms of the potential drop across *AH*. Hence when

[1] See Harrison and Foote, Jour. A. I. E. E., Feb. 20, 1920.

the instrument is connected through the line of resistance L to the couple of resistance T the rheostat CB must be adjusted until $r_1+L+T=r_5$. When this adjustment is made, the scale reading gives correctly the emf e of the couple or the correct temperature if the scale is graduated in degrees. This adjustment is made in the following manner: By depressing the key K, a portion of the galvanometer resistance r_2 is short-circuited and the rest of the resistance r_3, containing the moving coil, is shunted by the resistance r_4. If e' represents the potential drop across DF when the key is open and e'' represents the potential drop when the key is closed, we obtain:

$$e'=\frac{er_3}{L+T+r_1+r_2+r_3} \tag{1}$$

$$e''=\frac{er_3r_4}{(L+T+r_1)\ (r_3+r_4)+r_3r_4} \tag{2}$$

If r_1 is so adjusted that these two potential drops and hence the deflections of the indicator are the same, we have on equating (1) and (2):

$$L+T+r_1=\frac{r_2r_4}{r_3}=\text{a constant.} \tag{3}$$

If, now, in the construction of the instrument $\frac{r_2r_4}{r_3}$ is made equal to r_5, the above setting makes $L+T+r_1=r_5$ the adjustment required in order that the reading of the scale give the true emf of the couple.

The ease with which the proper setting can be obtained is greatly improved by making $\frac{r_3}{r_4}$ equal to from 5 to 10. Suppose $\frac{r_3}{r_4}$ is made equal to 9. Then, if r_1 is improperly adjusted and the instrument reads in error by say δe when the key K is open, on depressing the key the reading is changed by $9\delta e$. If now r_1 is readjusted with the key depressed until the reading takes its initial value, the error with the key open is reduced to $\frac{\delta e}{10}$. The process for operating the instrument is accordingly as follows:

1. Read the instrument with the key open.

2. Close the key and adjust the rheostat r_1 until the instrument reads approximately the same as in 1.

3. Repeat 1 and 2, if necessary.

When $\frac{r_3}{r_4} = 9$ it is rarely necessary to make a second adjustment. In position 1 the instrument acts as an ordinary galvanometer. The single setting in position 2 reduces the error in the ordinary galvanometer by the factor $\frac{1}{10}$, which is usually sufficient. The adjustment for the proper external resistance, if desired, can be made with 10 times the precision necessary. Thus, if the galvanometer can be read to $\frac{1}{10}$ of a scale division, the line resistance can be set for an error less than $\frac{1}{100}$ of a scale division, which is at least 10 times the accuracy possible with any indicating instrument. Hence, the factor of variable line resistance which may give rise to very serious errors is easily and accurately controlled by a simple mechanical adjustment.

The device is readily applicable to multiple installations of different line resistance. For multiple-point recorders and indicators as many resistances *BC* may be employed as there are couples. These may be inexpensive rheostats having a resistance of approximately 15 ohms each located in each couple line between the couple and the selective switch. These rheostats may be adjusted in the manner described whenever convenient or necessary. The following illustrates a suitable proportioning of resistance for a 300-ohm indicator:

$$r_2 = 135 \text{ ohms.}$$
$$r_3 = 150 \text{ ohms.}$$
$$r_4 = 150/9 \text{ ohms.}$$
$$r_5 = 15 \text{ ohms.}$$
$$r_2 + r_3 + r_5 = 300 \text{ ohms.}$$

If the simple indicator has the proper ratio of manganin to copper, its temperature coefficient is practically zero. In that case the shunt r_4 should have the same manganin to copper ratio as the portion of the galvanometer resistance comprised by r_3. The complete instrument will have, accordingly, a zero temperature coefficient. If the simple indicator does not have a zero temperature coefficient, it is possible by increasing the proportion of manganin in r_4 to compensate for the temperature coefficient of the resistance r_3 so that the complete instrument has a zero temperature coefficient.

Fig. 21*b* shows another wiring diagram for an instrument which accomplishes the same results as the one already described. For many purposes this is the more desirable arrangement, since

resistance at the contact point in the switch can produce no error, and if the instrument is properly damped with the key open it will be also properly damped with the key closed.

When the variable resistance r_1 is so adjusted that the position of the pointer is unchanged by closing key K, the following relation exists between the resistances:

$$r_5 = \frac{r_2 r_4}{r_6}, \text{ where } r_5 \text{ is defined as before.}$$

This adjustment is, therefore, independent of the value of r_3, which may be altered slightly, when desired, for calibrating the instrument. It is also independent of the resistance of branch CK, and therefore of resistance in the key.[8]

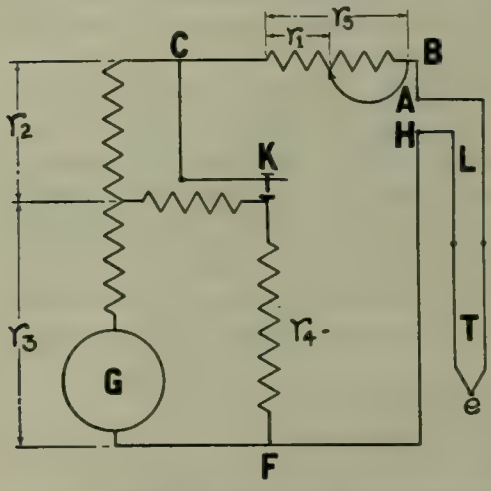


FIG. 21*b*.—*Another wiring diagram employing the Harrison-Foote method*

When resistances r_2 and r_4 are made of manganin and r_6 of copper or other material with high temperature coefficient β, it is seen that as r_6 increases r_5 must be decreased if the instrument is to read the same with the key open or closed.

Let α = temperature coefficient for current when the instrument is kept adjusted at all temperatures.

Let R_g = resistance of galvanometer coil and springs.

Let γ = ratio of temperature coefficients of resistance of R_g and r_6.

Let i' = current through galvanometer required for full scale deflection.

Let e' = desired emf reading corresponding to full scale deflection.

Let $S = \frac{e'}{i'}$.

Let R_d = total resistance of galvanometer circuit necessary for critical damping.

Let K = multiplication factor for errors (represented by $\frac{r_3}{r_4}$ in foregoing discussion).

[8] Harrison and Foote, Jour. A. I. E. E., Feb. 20, 1920.

The following relations exist between the various characteristic constants and resistances:

$$r_3 = \left[\frac{\alpha}{\beta} - \frac{r_5}{S} + \frac{R_g \gamma}{R_d}\right] \frac{S\ R_d}{S - R_d}$$

$$r_2 = \frac{S}{R_d}(S - r_3) - r_5$$

$$K = \frac{r_2}{r_5} \frac{S - R_d}{S - r_3}$$

$$r_4 = r_5 \frac{R_d}{S - R_d}$$

$$r_6 = \frac{r_2 r_4}{r_5}.$$

For zero temperature coefficient, γ is made equal to the combined temperature coefficients of the springs and magnet; that is, γ is taken about -0.0002.

If a very low range instrument is being designed, and it is not readily possible to realize a zero temperature coefficient, a larger negative value is chosen for γ. R_g can never be larger than r_3.

For a multiple range instrument r_3 may be made of a number of resistances, any of which may be short-circuited by a suitable arrangement. Without additional wiring, however, the instrument would not be critically damped for more than one range.

Since compensation is made for all variations of resistance, this instrument may be used as the galvanometer of a simple type of potentiometer, and an accurately compensated deflection potentiometer will be obtained.[9]

(*b*) Potentiometer Method.—The most accurate method for measuring the emf of a thermocouple is by use of a potentiometer. The fundamental principle of this instrument is illustrated by Fig. 22. A constant current from the battery B flows through the slide wire resistance abc. One wire of the couple T is connected to the movable contact b and the other wire, in series with a sensitive galvanometer, is connected to a. The contact b is moved until the galvanometer reads zero, thus showing that no current is flowing through the thermocouple circuit. When this balance or zero setting is made, the true emf of the couple is equal to the potential drop across ab. This is obtained from Ohm's law, $e = i\,r$, where i is the current flowing through the resistance $r = ab$. If the current is always adjusted to a definite value the

[9] Harrison and Foote, Jour. A. I. E. E. Feb. 20, 1920.

slide wire may be graduated to read millivolts or temperature directly. Various devices are employed for this purpose. An ammeter will serve, but the usual method is to obtain a preliminary setting by replacing the thermocouple by a constant known source of emf, such as a standard cell. The galvanometer *G* is always used as a zero instrument in a strictly potentiometric circuit. Hence, it requires no calibrated scale, and no attention need be given to the constancy of its sensitivity, provided it is always sufficiently sensitive to serve its purpose. These requirements are easily met, and the entire potentiometer, galvanometer, battery, standard cell, slide wires, etc., as constructed are mounted in a case not much larger than that of a millivoltmeter. The instrument is as mechanically robust as indicators of equal sensitivity operating on the galvanometric principle.

There are several important advantages in the potentiometer method. The emf or temperature scale is easily made very open, thus permitting accurate readings. The calibration of the scale is in no way dependent upon the constancy of magnets, springs, or jewel bearings, nor upon the level of the instrument. From the pyrometric standpoint, however, the greatest advantage is the complete elimination of any error due to ordinary changes in the resistance of the couple or of the lead wires. Thus if a potentiometer is correctly balanced and the resistance of the thermocouple is then greatly increased, the balance is unchanged. However, excessive resistance in the couple circuit reduces the sensitivity of a setting. Thus if the resistance is very high, there appears to be a considerable range over which the balance is satisfactory. This range can be decreased by increasing the sensitivity of the galvanometer, but for obvious reasons this is already made as high as is consistent with sufficient ruggedness.

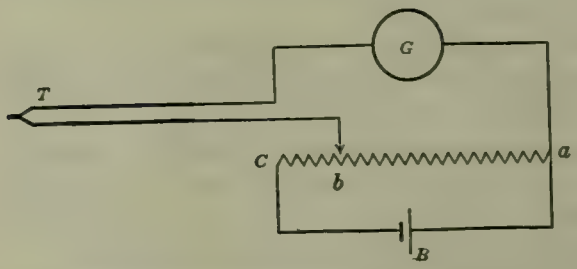


FIG. 22.—*Simple wiring diagram for potentiometric indicator*

The sensitivity of the galvanometer also determines the proper length of the emf scale. Thus there is little advantage gained in making the scale or slide wire of such length that a millimeter deflection on the galvanometer scale corresponds to more than a

few millimeters on the emf scale. The emf scale of one type of portable instrument has a length of 40 cm, while in another the scale is 250 cm long.

The only objections to the potentiometer are slightly greater initial cost and the fact that usually a manual adjustment must be made to obtain a setting. In the potentiometric recording instrument, however, all the various manipulations may be performed automatically, even to the balancing against the standard cell.

Fig. 23 illustrates the Leeds & Northrup Co. portable potentiom-

FIG. 23.—*Type of portable potentiometer*

eter and Fig. 24 shows the wiring diagram. The galvanometer should be adjusted to read zero on open circuit. At intervals of a few hours the setting on the standard cell should be made. This is done by depressing the key *S.C.* and adjusting the resistance *RR'* by turning the knurled head on the left of the case until the galvanometer reads zero. The thermocouple is connected to the terminals *T.C.*, and the emf or temperature is obtained directly from the dial scale by depressing the key marked *T.C.* and turning the main dial until the galvanometer indicates zero. The key *T.C.* and the key *S.C.* should never be depressed

at the same time. These instruments can be made in any scale range or with multiple scale ranges adapted to various types of couple.

The Brown portable potentiometer has a scale length of 96 inches, graduated in 1/50 millivolt intervals. The galvanometer, which is of the pivot type, has a higher sensitivity than the suspension instruments usually employed in portable potentiometers.

(*c*) SEMIPOTENTIOMETER METHOD.—It is possible by means of a single galvanometer or millivoltmeter to measure the emf of a couple potentiometrically. Thus, in Fig. 22, by using a shunted galvanometer first in the main circuit *abcB* as an ammeter, the initial setting for a standard current is obtained. Then the instrument without the shunt is placed in the position *G* and the contact *b* is moved along the graduated slide wire until a zero setting is obtained. The objection to this method is that, if the millivoltmeter is sufficiently sensitive to be used as a zero instrument, it may not be reliable as an ammeter, and if reliable as an ammeter it may not possess sufficient sensitivity when used as a detector or null instrument. The semipotentiometer method, however, in other forms has proven of value in thermoelectric pyrometry.

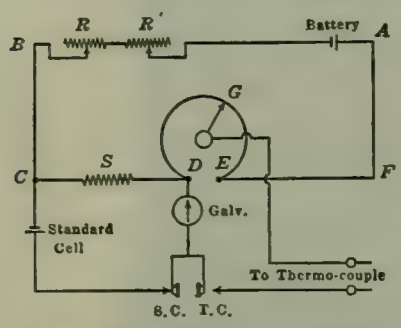


FIG. 24.—*Wiring diagram of potentiometer shown in Fig. 23*

(1) *Northrup's Method.*—Fig. 25 shows a wiring diagram of the pyrovolter, an instrument based on Northrup's method. In position (*a*) the dry cell *B* contained in the case of the instrument sends a current through the variable resistance *R* and the fixed resistances *C* and *S*. The resistance *C* of copper is equal in value to the resistance of the copper coil of the moving element of the galvanometer *G*. The couple *T* is connected in series with the moving coil of the galvanometer across the resistance *S*. The resistance *R* is adjusted until the galvanometer reads zero by turning the knurled head on the lower right-hand corner of the instrument. The key on the lower left-hand corner is then depressed, which throws the thermocouple and the resistance *C* out of the circuit and replaces *C* by the galvanometer *G* of equiva-

lent resistance, Fig. 25 (*b*). The galvanometer is now deflected by an amount proportional to the current flowing through it, which in turn is proportional to the potential drop across *S*. The scale of the instrument is graduated to read the potential drop over *S*, and, since this potential difference was made equal to the emf of the couple by the initial setting for zero deflection, the galvanometer indicates directly the true emf of the couple. The initial setting is not altered by introducing resistance into the thermocouple circuit, so that the instrument is really a form of potentiometer. The scale may be graduated to indicate temperature for any particular type of couple, and the instrument may be obtained with several different scale ranges.

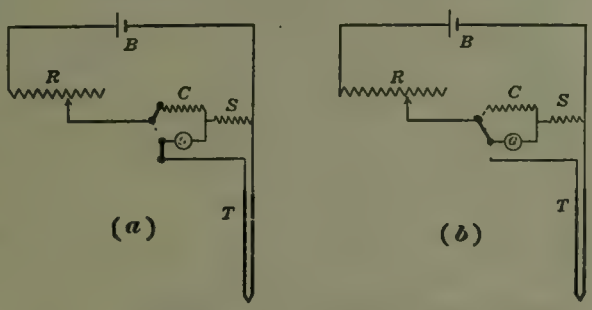


FIG. 25.—*Wiring diagram illustrating Northrup's method*

(*2*) *Northrup's Method Applied to Continuously Deflecting Instruments.*—This instrument is the ordinary pyrovolter with the addition of an extra key and an adjustable resistance. After the emf of the couple has been determined by the pyrovolter method just described, the galvanometer in series with this resistance is connected directly to the thermocouple terminals. The resistance is then adjusted until the reading of the instrument is the same as that determined by means of the pyrovolter.

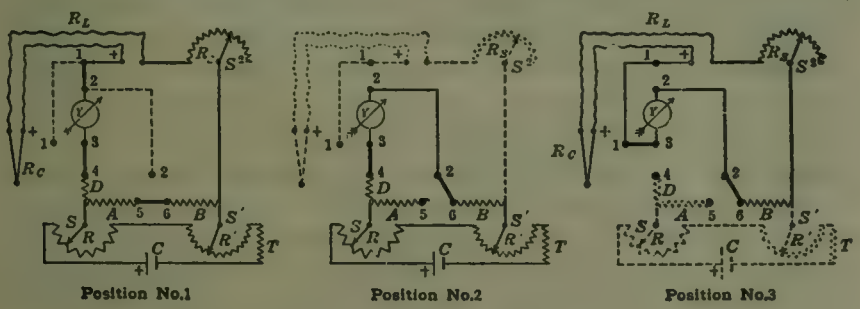


FIG. 26.—*Wiring diagram illustrating continuously deflecting instrument embodying a method described in the text*

Figure 26 is the wiring diagram of another instrument known as the Brown precision heatmeter which, making use of somewhat

differently arranged circuits, employs a principle very similar to that of the Northrup continuously deflecting pyrovolter. By means of suitable switches the electrical connections are thrown successively into the three positions illustrated, position No. 3 being the final working position, in which the couple is connected directly to the millivoltmeter through a definite line resistance. In position No. 1 the emf of the couple is balanced against the potential drop between S and S' by varying R and R' until the galvanometer Y indicates zero. In position No. 2, the thermocouple circuit is cut out and the galvanometer is connected to the points S and S'. The scale of the galvanometer is so divided as to read the potential differences across SS'. This potential difference is not altered by switching from position No. 1 to position No. 2, since the resistance D is so chosen that $A = Y + D$. The total resistance of the galvanometer circuit in position No. 2 is $D + Y + B$. In position No. 3 the resistance R_s is adjusted until the total resistance of the galvanometer circuit is equal to that of position No. 2, viz.:

$$D + Y + B = R_c + R_L + R_s + B + Y$$

This adjustment is obtained when the reading of the galvanometer is not altered by switching from position No. 2 to position No. 3. Thus with R_s properly adjusted, the reading of the indicator in position No. 3 gives directly the true emf or the temperature of the couple as long as the line resistance $R_L + R_c$ remains unchanged.

(*d*) *Deflection Potentiometer Method.*—A potentiometer is ordinarily used as a null instrument. The emf of the couple is exactly balanced by the potential drop over a resistance through which a current from a battery is flowing. No current flows through the couple circuit when the proper setting is obtained. This condition is indicated by zero deflection of the galvanometer in series with the couple. The objection sometimes raised against the ordinary potentiometer for industrial installations is that it requires a manual adjustment of a dial or slide wire every time an observation is made. This objection is practically eliminated in the deflection potentiometer, which may be constructed so that it embodies the accuracy of the ordinary potentiometer and the convenience of the galvanometer indicator.

In the deflection potentiometer, part of the emf of the couple is balanced against the potenial drop over a resistance through which a current is flowing, and the remaining portion of the emf is indicated by the deflection of the galvanometer in series with

the couple. For example, the instrument may be constructed with a dial of say 16 points, representing potential differences from 0 to 15 millivolts, and a galvanometer which gives full scale deflection on 1 millivolt. The dial is set to the approximate emf developed by the couple, and the dial reading combined with the galvanometer reading gives the true emf of the couple. If the couple developed an emf of 13.58 millivolts, the value 13 is obtained from the dial and the value 0.58 is read from the galvanometer scale. Usually the scale is graduated both ways with zero at the center, so that + or − deflections may be readily shown. Thus, in the above example, if the dial had been set at 14 the pointer would have read -0.42, and the final reading would be $14.00-0.42=13.58$, as before. To avoid the necessity of thus subtracting a number from the dial setting, the galvanometer scale may be graduated from left to right as follows:

0, 10, — 80, 90, 0, 10, 20 — 100

When the pointer swings to the left, the next lower dial reading is used. Hence in the above example, with the dial set at 14, the galvanometer would read 58 on the left part of the scale. Thus the correct value is 13.58 as before. In many industrial processes the temperature of the couple will vary only slightly during several hours, so that a new dial setting is infrequently required. Thus the method for obtaining the emf of the couple is nearly as simple as when an ordinary galvanometric indicator is used.

The theory of the deflection potentiometer has been developed in detail by Brooks.[10] As applied to emf measurements the simple theory may be deduced as follows: It has been shown in the case of an ordinary potentiometer that if an emf e' is exactly balanced against the potential drop in a resistance wire of a potentiometer, the value of this emf will be given by the equation

$$e'=\frac{Er_1}{r_1+r_2}, \qquad (1)$$

where E is the emf of the battery used to furnish the current in the resistance wire, r_1 is the resistance of this wire, and r_2 is all other resistance in the battery circuit. Usually the value of e' is indicated by figures on the dials or slide wire of the potentiometer. If the emf e' changes to a new value e and the potentiometer

[10] B. S. Sci. Papers, Nos. 33, 79, 172, and 173.

remains adjusted as before, a current will flow through the galvanometer and thermocouple, causing the galvanometer to deflect. The currents now flowing through r_1 and r_2 are unequal and are different from the original value. The currents flowing in the different branches of the circuit are indicated by Fig. 27, in which T represents a thermocouple, G the galvanometer, $abcB$ the potentiometer, R the resistance of the galvanometer and thermocouple, e the emf of the couple, and B the battery. I represents the current flowing from a to b through r_1 and i the current flowing through the galvanometer. Since the sum of the emfs and potential drops around any closed circuit must equal zero, the following two equations may be written:

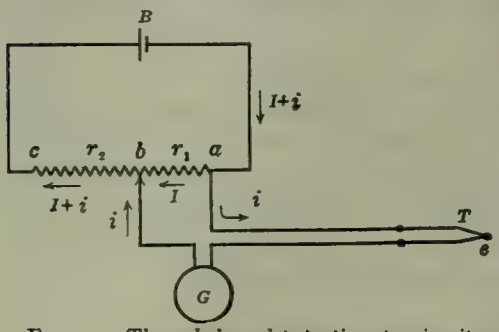


FIG. 27.—*The unbalanced potentiometer circuit*

$$E = (r_1 + r_2)I + r_2 i \tag{2}$$

$$e = r_1 I - Ri \tag{3}$$

Substituting the value of I from equation (2) in equation (3) and subtracting this value of e from the value of e' given by equation (1) we obtain

$$e' - e = i\left(R + \frac{r_1 r_2}{r_1 + r_2}\right),$$

or

$$i = \frac{e' - e}{R + \frac{r_1 r_2}{r_1 + r_2}}. \tag{4}$$

Hence when a potentiometer is not balanced a current will flow through the galvanometer equal to the difference between the setting of the potentiometer and the emf of the thermocouple, divided by the total resistance $R + r_1 r_2/(r_1 + r_2)$ in the galvanometer circuit.

Representing the resistance within the potentiometer $r_1 r_2/(r_1 + r_2)$ by r_p, the resistance of the galvanometer circuit will equal $R + r_p$. If the galvanometer is to indicate correctly at all values of e' the unbalanced emf $e' - e$, its sensitivity must remain constant. This requires that $R + r_p$ remain constant. The value of r_p will change as point b is moved nearer to a or c, thus altering the values of r_1 and r_2. Hence, in order that $R + r_p$ remain constant it is necessary

to put in the galvanometer circuit a variable resistance which compensates for these changes in r_p.

In instruments of low range, suitable for thermocouples, r_1 is made small compared with r_2. Since $r_p = \frac{r_1 r_2}{(r_1 + r_2)}$, if r_2 is sufficiently large compared with r_1, we may neglect the term r_1 in the denominator, and the above equation reduces to $r_p = r_1$. For such an instrument the compensating resistance in series with the galvanometer is decreased by the value of r_1 at any dial setting.

Instruments of this type have been designed by Brooks and by White. The compensating resistance is mounted as an integral part of the dial, so that turning the dial changes the emf setting and at the same time adjusts the compensating resistance in the galvanometer circuit to its proper value.

Fig. 28 illustrates the wiring diagram of a deflection potentiometer for thermocouples, known as the Taylor Instrument Co.'s "range control board." The galvanometer G is provided with two scales, in the ranges zero to 500° C and 450 to 950° C, respectively. The galvanometer circuit is connected at fixed points a and b within the potentiometer, and when the instrument is to operate in the lower range the battery circuit is opened. Thus the potentiometer setting e' is made zero without changing the value of r_1. In this range the instrument operates as an ordinary galvanometric indicator. If the temperature of the thermocouple is above 450 or 500° C, a current of such magnitude is made to flow through r_1 that the potential drop e' across r_1 balances the emf developed by the couple when at 450° C. The temperature will then be indicated on the high range scale.

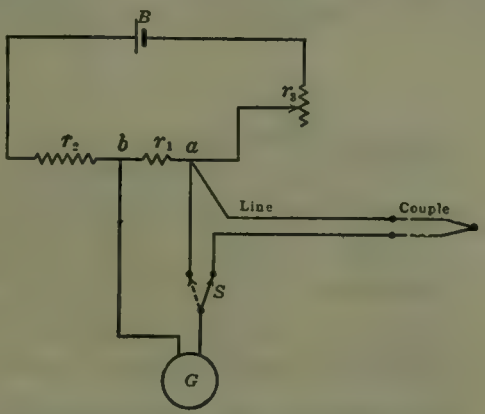


FIG. 28.—*Wiring diagram of a deflection potentiometer*

The total resistance of the galvanometer circuit is almost exactly equal when operating in either range, since when operating in the upper range the shunting effect of $r_2 + r_3$ on r_1 is negligible. Since a separate galvanometer scale is provided for each setting of the potentiometer (o and e') it is not really necessary that the sensitivity be equal in the two cases.

For the high range, the current from the battery is adjusted by connecting switch S, as shown by the dotted line, and setting r_3 so that the galvanometer deflects to a marked position. Provision must be made for reversing the direction of the current from the battery through the galvanometer after this adjustment has been made. The figure does not show this or the switch for opening the circuit when the instrument is to be used for the low range.

Fig. 29 is the wiring diagram of the Leeds & Northrup deflection instrument for thermocouples. When a range suitable for thermocouples is used and the condition of a balanced Wheatstone bridge with arms of equal resistance is never far departed from, the resistance of that part of the galvanometer circuit which is constituted by the potentiometer usually remains constant within a few tenths of 1 per cent. The value of this resistance for instruments of the same range and using the same battery current is considerably higher than that of the designs discussed above.

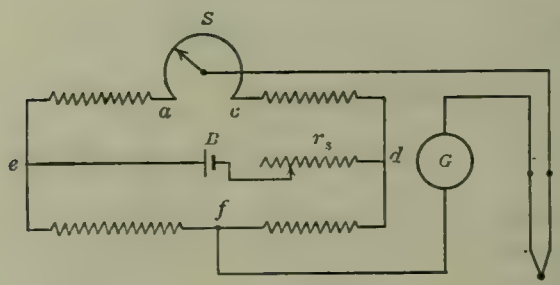


FIG. 29.—*Wiring diagram of a deflection potentiometer*

The following table shows suitable values of the different resistances which will give to a satisfactory degree, conditions of a nearly balanced Wheatstone bridge with equal arms:

Resistance e to a=475 ohms.
Resistance a to c= 70 ohms.
Resistance c to d=455 ohms.
Resistance e to f=475 ohms.
Resistance f to d=525 ohms.
dBe may vary from zero to infinity.

These values may be divided or multiplied by any number in order to obtain any desired range of currents and a proper critical damping resistance for the galvanometer. The above design allows a range of 70 millivolts over the slide wire. The curves in Fig. 30 show the manner in which the resistance r_p of that part of the galvanometer circuit consisting of the parallel paths within the potentiometer varies with different settings of the slide wire, ranging from zero to 70 millivolts, and with resistance r_3 in the outside battery circuit dBe, varying from zero to infinity.

Actually r_3 would not be likely to vary beyond the limits 100 and 500 ohms. An average value for r_p may be obtained by setting the slide wire to read about 7 millivolts, and the variation of r_p from this value will be, in general, less than 0.1 per cent, whatever the setting of the slide wire on the battery resistance r_3. Therefore, if the galvanometer is calibrated when the slide is set to read 7, it will be more nearly accurate with varied settings and adjustments of the instrument. Since r_p constitutes only part of the galvanometer circuit, the galvanometer sensitivity will remain constant within proportionally less than 0.1 per cent.

This instrument is designed primarily so that the slide wire may be set to read the exact temperature required. The galvanometer G accordingly indicates the departure of the actual temperature from the temperature desired. It thus serves as a very convenient guide to the operator of a furnace, who can see at a glance by how many degrees the temperature at any time differs from the temperature at which the furnace should be operated.

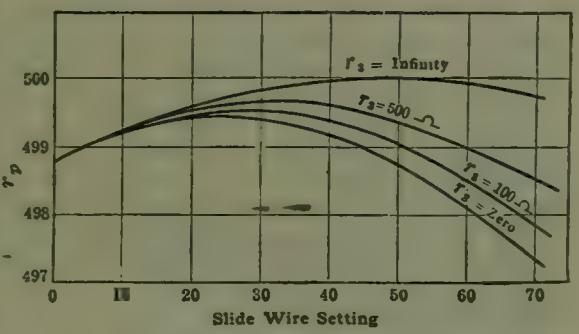


FIG. 30.—*Illustrating change of r_p with slide wire setting in the potentiometer illustrated in Fig. 29*

The Cleveland Instrument Co. deflection potentiometer is a modification of their Wheatstone bridge cold-junction compensating instrument. By altering the ratio of the coils, Fig. 37, in suitable steps, the emf of the couple is opposed by potential drops of different values, the indicator showing the unbalanced portion of the thermocouple emf, as in the other instruments described.

(1) *Deflection Potentiometer Graduated to Read Temperature Directly.*—The above discussion has assumed that the scale of the galvanometer may be graduated to read either emf or temperature. If the thermocouple has a linear relation between emf and temperature, the theory outlined is just as applicable to a scale and dial graduated in terms of temperature as in terms of emf. For example, the dial may consist of 13 points, representing 0, 100, 200, ... 1200° C, and the galvanometer scale may be graduated

from 0 to 100° C for the intermediate temperatures. Thus a temperature of 1124° C would be indicated by a dial setting of 11 and a scale reading of 24. If the temperature-emf relation of the couple is not linear, a given temperature change corresponds to different changes in emf, depending upon the temperature of the couple. Hence, in order that the scale of the galvanometer may be graduated in degrees, it is necessary to modify its sensitivity in the various temperature ranges by means of series resistance. Thus, instead of maintaining the total resistance, $R + r_p$, of the galvanometer circuit constant, as in the case of the deflection potentiometer graduated in terms of emf, this resistance must be so adjusted that the change in sensitivity of the galvanometer compensates for the change in thermoelectric power of the couple with temperature. The change in thermoelectric power of the couple over the temperature range represented by the scale of the galvanometer is usually small, and hence causes only slight errors.

In the "range control board" the above-mentioned difficulty is avoided by the provision of a separate scale for each range; but it would not be practical to apply this method to the ordinary deflection potentiometer, which may have 10 to 20 different scale ranges. In the Leeds & Northrup deflection potentiometer, Fig. 29, the ratios of the resistances may be so modified as to produce very closely the proper compensation in r_p for any type of couple used industrially.

(*e*) Temperature of the Cold Junctions of Thermocouples.—The emf developed by a thermocouple depends upon the temperature of the cold junctions, as well as upon the temperature of the hot junction. For certain base-metal couples having a nearly linear relation between temperature and emf, the emf is approximately proportional to the difference in temperatures of the hot and cold junctions. With such couples a change of 50° C in the temperature of the cold junctions, unless corrected for, would result in an error of 50 °C in the final temperature measurement.

If a couple is calibrated with a cold-junction temperature of t_o°C and used with a cold-junction temperature of t'_o°C, the true temperature of the hot junction is obtained by adding to the observed temperature the value $(t'_o - t_o)\ K$ where K is a factor depending upon the particular couple employed and upon the temperatures of the hot and cold junctions. The factor K varies from 1.5 to 0.3, but for rough work may be assumed to be 1.0 for base-metal couples and 0.5 for rare-metal couples.

The following table gives the cold-junction factors for several different types of couple:

TABLE 9.—Cold-Junction Correction Factors

Engelhard "Le Chatelier"		Johnson-Matthey "Le Chatelier"		Copper-constantan		Iron-constantan		Chromel-alumel	
Temperature	K^a	Temperature	K^a	Temperature	K^a	Temperature	K^a	Temperature	K^a
°C		°C		°C		°C		°C	
250–450	0.65	250–400	0.60	0–50	1.00	0–100	1.00	0–800	1.00
450–650	.60	400–550	.55	50–80	.95	100–600	.95	800–1100	1.05
650–1000	.55	550–900	.50	80–110	.90	600–1000	.85		
1000–1450	.50	900–1450	.45	110–150	.85				
				150–200	.80				
				200–270	.75				
				270–350	.70				

[a] Based on calibration with t_0=0°C.

The corrections may be applied directly, without computing, by setting the pointer of the galvanometer to read the cold-junction temperature on open circuit. The setting is made by turning the zero-adjustment screw of the indicator when the couple is disconnected. This method of correcting for the cold-junction temperature is accurate, but requires, of course, new settings whenever the temperature of the cold junction is altered. The Bristol compensator performs this adjustment automatically by means of a bimetallic spring connected to one of the control springs of the moving coil. The cold junctions of the couple must be accordingly located at the instrument either by direct connection or by means of extension leads, described later. Indicators of the potentiometric type frequently have a movable slide on the temperature scale or an auxiliary dial (see discussion below) which when set to the temperature of the cold junction gives perfect compensation at all temperatures of the hot junction. These two compensation methods also require new settings whenever the temperature of the cold junction is altered. With large and permanent installations the applying of corrections for the temperature of the cold junction by any of the above methods is frequently troublesome, since the temperature may vary considerably in practice over a few hours. There are several methods for obviating the necessity of applying such corrections and of making manual adjustments. The head of the couple may be fitted with a water jacket through which water flows at practically constant temperature from the water main. Copper wires lead from the ter-

minals of the couple inside the water jacket to the indicator, and the pointer of the indicator on open circuit is set to read the mean temperature of the water, usually 10 to 20 °C. If a potentiometer indicator is used, the zero adjustment slide or the cold-junction dial is set at this temperature.

(1) *Compensating Leads.*—The use of compensating lead wires from the couple to the indicator is, in general, the most satisfactory method of minimizing the cold-junction errors in industrial installations. For base-metal couples these lead wires are of nearly the same materials as those employed in the couple. For example, with a chromel-alumel couple one lead wire is chromel, which is connected to the chromel wire of the couple. The other lead wire of alumel is connected to the alumel wire of the couple. Small stranded wires are used for flexibility. Thus, the cold junction is carried away from the head of the couple, where the temperature varies, to a point at some distance from the furnace, where the temperature is reasonably constant, and from this point copper wires lead to the indicator. The compensating wires may terminate in a thermostated cold-junction box, as illustrated by Fig. 31, or may be buried under ground. At a depth of 10 feet beneath the floor of a large building the tempera-

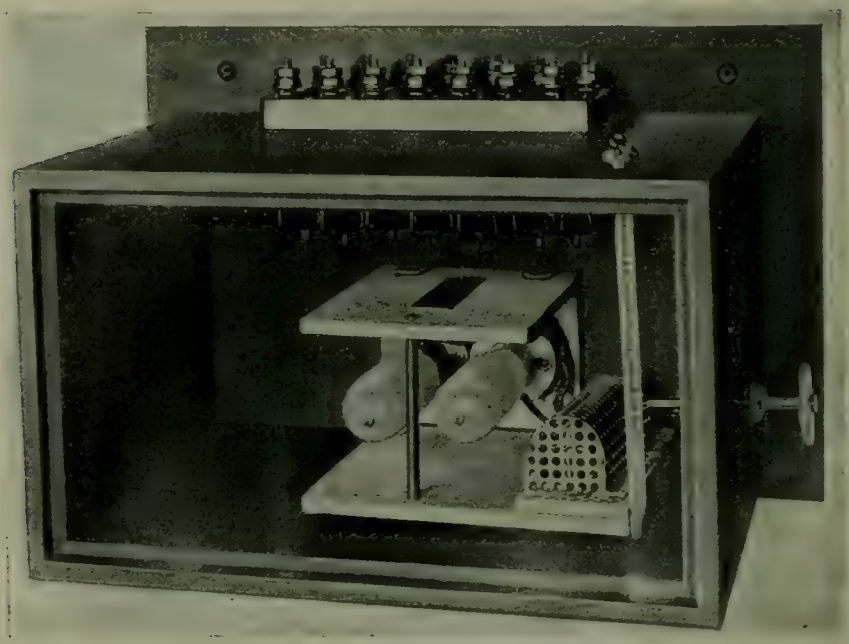


FIG. 31.—*Thermostated cold-junction box*

ture remains constant to within 2° C throughout the year. Usually this mean temperature is about 12° C for temperate climates, but may differ from this value somewhat if the location is in the immediate vicinity of a large furnace. To use this method for controlling the temperature of the cold junctions, an iron pipe of the proper length, closed at the bottom, is driven into the ground, and the two cold junctions—for example, copper-alumel and copper-chromel, well soldered and carefully insulated—are threaded to the bottom of the pipe in such a manner as to be conveniently removable when necessary. The top of the pipe may be plugged with asbestos or waste and covered with pitch to keep water away from the insulation. The scale of the indicator is set to read the mean temperature of the bottom of the tube. It is convenient to have an extra pair of compensating leads or an extra thermocouple with its junction at the bottom of the pipe to measure this temperature occasionally. Usually the compensating leads of a base-metal couple are marked or are equipped with one-way terminals, so that they are easily connected properly to the head of the couple. If reversed at the couple, the leads will cause an error double the amount of the compensation. When compensating leads of a base-metal couple are properly connected to the couple, no deflection of the indicator is obtained by heating the head of the couple.

The high cost of platinum prevents the use of compensating leads of the same metal in the case of a rare-metal couple, but inexpensive lead wires of copper and an alloy of nickel-copper are now available for use with the platinum, platinum (90 per cent)-rhodium (10 per cent) couple (Bristol compensating leads). These lead wires do not compensate individually, but taken together they compensate to within 5° C for a variation of 200° C in the couple-lead wire junctions. Since the compensating lead wires for the rare-metal couple do not compensate individually, both terminals on the head of the couple should be always as nearly as possible at the same temperature. The copper wire of the compensating leads is connected to the platinum-rhodium wire of the couple, and the copper-nickel wire is connected to the platinum wire of the couple—that is, alloy wire to pure metal—in each case. The cold junction is then located at the indicator end of the compensating leads. The temperature of this junction may be controlled, if necessary, by one of the methods described above Copper wires are carried from this point to the indicator.

(2) *Potentiometric Compensation Methods.*—The wiring diagram for the Leeds & Northrup portable potentiometer equipped with a hand-adjusted, cold-junction compensator is given in Fig. 32. The emf of the thermocouple *H* is balanced against the potential drop across *DG*, a condition obtained when the galvanometer reads zero, as discussed previously in the section on potentiometric indicators. The dial *G* is calibrated to read the temperature of the hot junction of the couple. If this temperature remains constant and the temperature of the cold junction *T* increases, the emf of the couple decreases and the point *G* would have to be moved nearer *D* to obtain a balance. If this were done, however, the temperature indicated on the scale would be too low. Hence, instead of moving *G*, the contact *D* is turned nearer *G* by an amount depending upon the temperature of the cold junction. A portion of the slide wire *DGE*, containing the contact *D*, is mounted as a separate dial, empirically graduated, for any particular type of couple, to read the temperature of the cold junction. The pointer on this dial is set at the cold-junction temperature, by which setting the contact *D* is moved the proper amount for exact compensation. The balance is then made in the usual manner by adjusting the contact *G*. The temperature now indicated on the main dial is the correct temperature of the hot junction of the couple. The arrangement shown in Fig. 32, while illustrating the principle of this method of compensation, does not provide for emf measurements to zero emf. The temperature scale on the main dial must start at the highest temperature on the cold-junction dial. Fig. 33 illustrates the wiring diagram more usually employed. The cold-junction dial is in parallel instead of in series with the main dial. This method permits settings on the main dial to zero emf. The theory of the compensation is similar to the theory of the automatic compensator described in the following paragraph:

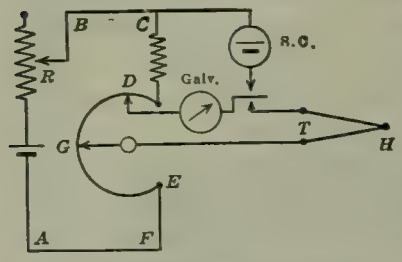


FIG. 32.—*Hand-adjusted cold-junction compensator*

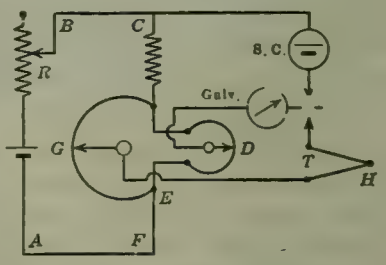


FIG. 33.—*Hand-adjusted cold-junction compensator*

The above principle has been applied in an automatic compensator which has been used satisfactorily with the Leeds & Northrup recorders, Fig. 34. The present circuit and the one just described are similar, with the exception that the contact D in Fig. 33 is mechanically fixed between the two resistances, M having a zero temperature coefficient of resistance and N of nickel having a high-temperature coefficient. The resistance N is located near the cold junction of the couple, so that its temperature and that of the cold junction are identical. If the temperature of the cold junction and of N increases, the emf developed by the couple decreases; but the accompanying increase in the resistance N automatically produces the same effect as moving the contact D toward G in Fig. 32. The circuit is more simply represented by Fig. 35. Let e = emf developed by the couple when the hot junction is at a temperature t°C and the cold junction o°C, and e_0 = the emf developed for a hot-junction temperature t_0 °C and a cold-junction temperature o°C. Then the emf developed by the couple when its hot junction is t°C and cold junction is t_0°C is $e - e_0$. Neglecting the slight effect of the variation of the resistance N with the temperature, the potential drop from K to E due to the battery B is constant $= e'$. Whence the potential difference between G and D when the galvanometer in the thermocouple circuit indicates zero is simply derived as follows and is equal to the emf of the couple:

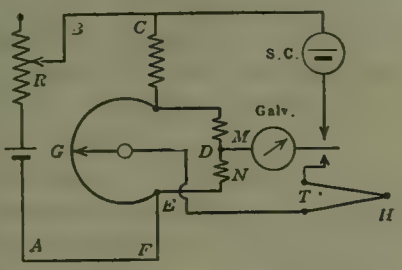


FIG. 34.—*Automatic cold-junction compensator*

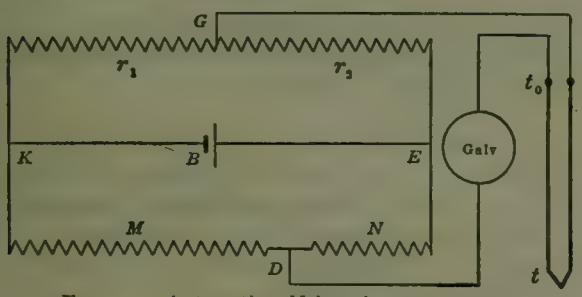


FIG. 35.—*Automatic cold-junction compensator*

$$\text{P. D. between } G \text{ and } D = e'\left(\frac{r_2}{r_1+r_2} - \frac{N}{M+N}\right) = e - e_0.$$

For exact compensation the position G must be independent of the cold-junction temperature t_0; hence on differentiating the above

expression we obtain the following as a condition which must be satisfied.

$$\frac{de_o}{dt_o} = e' \frac{M}{(M+N)^2} \frac{dN}{dt_o}$$

By properly proportioning N and M this condition is well satisfied for either base-metal or rare-metal couples.

(3) *Compensation by Use of a Shunt.*—The use of a resistance having a high temperature coefficient shunted across the terminals of the couple at the cold junction was suggested by Foote [11] in 1913 as a possible method of partially correcting for the cold-junction errors. A modification of this method has been since patented by Mertelmeyer [12], and is used by the Bristol Co. When the temperature of the cold junction of a couple increases the emf developed by the couple decreases. If, however, the resistance of the coil shunted to the couple increases with the temperature, the potential drop measured across the coil tends to increase, and by properly proportioning the various constants of the circuit a fair degree of compensation is obtained. Fig. 36 illustrates the method of compensation for potentiometric measurements.

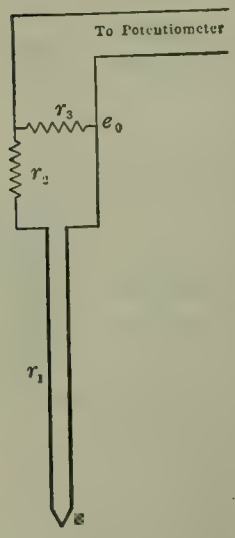


FIG. 36.—*Cold-junction compensation by shunt and series resistances*

Let r_1 = resistance of the thermocouple.
Let r_2 = series resistance having a zero temperature coefficient (manganin).
Let r_3 = resistance at 0°C of the shunt.
Let r'_3 = resistance of shunt at temperature t°C.
Let α = temperature coefficient of resistance of the shunt reckoned from 0°C.
Let β = thermoelectric power of the couple (assumed constant).
Let t = any temperature of the hot junction.
Let t_o = any temperature of the cold junction and of r'_3.
Let t' = temperature of hot junction for which perfect compensation is desired.
Let t_o' and 0°C = temperatures of cold junction for which perfect compensation is desired.

[11] Foote, B. S. Sci. Papers, No. 202, p. 12.
[12] Mertelmeyer, assignor to Bristol Co. U. S. Patent No. 1 228 803; 1917.

Let e' = potential drop across r'_3, hot junction = t°C, cold junction = t_o °C.

Let $e - e_o = \beta\,(t - t_o)$ = emf developed by couple.

In order to reduce the effect of the variation in the resistance r_1 of the couple to a minimum, a relatively high resistance r_2 of manganin is mounted in series with the couple as illustrated. If r_2 is sufficiently high in comparison with r_1, we may neglect consideration of the latter in the present discussion. The potential drop e''' across r'_3 for a cold-junction temperature t'_o °C and a hot-junction temperature t'°C is as follows:

$$e''' = \frac{\beta(t' - t'_o)\ (1 + \alpha t'_o) r_3}{r_2 + r_3(1 + \alpha t'_o)} \tag{1}$$

For a cold-junction temperature t_o = o°C and a hot-junction temperature = t' °C the potential drop e'' is given by equation (2):

$$e'' = \frac{\beta t' r_3}{r_2 + r_3} \tag{2}$$

For exact compensation e'' must equal e'''. Hence on equating (1) and (2) we obtain (3) as a conditional equation.

$$\frac{r_3}{r_2} = \frac{\alpha(t' - t'_o) - 1}{1 + \alpha t'_o}. \tag{3}$$

For a cold-junction temperature = t_o °C and a hot-junction temperature = t° C the potential drop across r'_3 is as follows:

$$e' = \frac{\beta(t - t_o)(1 + \alpha t_o) r_3}{r_2 + r_3(1 + \alpha t_o)}. \tag{4}$$

For a cold-junction temperature = o° C and a hot-junction temperature = t° C the potential drop across r'_3 is given by equation (5).

$$e'''' = \frac{\beta t r_3}{r_2 + r_3}. \tag{5}$$

The error in the compensation, expressed in degrees, at any hot-junction temperature t and cold-junction temperature t_o is given by equation (6).

$$\text{Error, in degrees} = \frac{e'''' - e'}{e''''} t = t - \frac{\left(1 + \frac{r_3}{r_2}\right)(t - t_o)(1 + \alpha t_o)}{1 + \frac{r_3}{r_2}(1 + \alpha t_o)} \tag{6}$$

On substituting the value of $\frac{r_3}{r_2}$ given by equation (3) one obtains:

$$\text{Error, in degrees} = t_o\left(\frac{t' - Kt}{t' - Kt_c}\right) \tag{7}$$

where

$$K = \frac{1 + \alpha t'_o}{1 + \alpha t_o}. \tag{8}$$

Suppose an iron-constantan couple is employed, and perfect compensation is desired for a hot-junction temperature of 800° C when the cold-junction temperature is 0° C or 50° C. The potentiometer indicator is graduated to read correctly all temperatures of the hot junction when the temperature of the cold junction is 0° C.

If the shunt is constructed of nickel wire we have the following data:

$\alpha = 0.006$
$\beta = 0.05$ millivolts per degree
$t' = 800°$ C
$t'_0 = 50°$ C

On substituting these values in equations (3) and (7) we obtain:

$$\frac{r_3}{r_2} = 2.7 \tag{9}$$

$$\text{Error, in degrees} = t_0\left(\frac{800 + 4.8t_0 - 1.3t}{800 + 3.5t_0}\right). \tag{10}$$

Equation (9) states that the ratio of the values of the resistance of the nickel shunt at 0° C and that of the manganin should be 2.7. Equation (10) gives the error in degrees for any temperature of the hot junction t and of the cold junction t_0. The following table shows the magnitude of these errors:

TABLE 10.—Error in Compensation by Shunt Method

Temperature of cold junction, degrees centigrade	Error with the hot junction at—		
	700° C	800° C	900° C
	°C	°C	°C
0	±0.0	±0.0	±0.0
10	−0.7	−2.3	−3.9
20	−0.3	−3.3	−6.3
30	+1.1	−3.2	−7.5
40	+3.5	−2.0	−7.6
50	+6.7	±0.0	−6.7
60	+9.7	+2.6	−4.5

Thus, over a variation of 200° C in the temperature of the hot junction and of 60° C in the temperature of the cold junction the method compensates to within 10° C.

The above method may be applied even more satisfactorily when a galvanometer is used instead of a potentiometer. By carrying through a series of computations for the more complicated circuit with a galvanometer of resistance R, a relation may

be obtained between R, r_2, r_3, α, and β. The following values give slightly better compensation around 800° C than that illustrated in the table above:

TABLE 11.—Shunt Compensation with Galvanometric Indicator

Galvanometer resistance	Series resistance (manganin)	Shunt resistance (nickel)
Ohms	Ohms	Ohms
100	125	150
100	59	100
100	23	50

It is evident that this method of compensation has certain advantages in the control of the temperature of a furnace and in maintaining this temperature at an approximately constant value. It should not be employed when the variation in temperature of the furnace is much greater than 100° C. There are few processes in which the use of compensated leads and a cold-junction box or a buried cold junction, as described above, is not to be preferred. The shunt method has been described at some length, however, because a discussion of it has never appeared elsewhere, and in certain restricted applications the method offers desirable features.

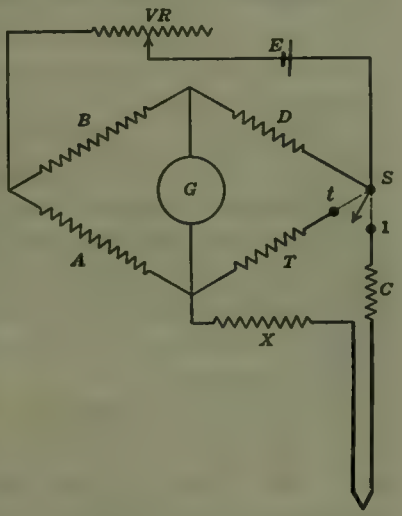


FIG. 37.—*Cold-junction compensation by Wheatstone bridge method*

(4) *Wheatstone-Bridge Compensation.*—This method of compensation for the temperature of the cold junction as applied by the Cleveland Instrument Co. is illustrated in Fig. 37. The switch S is first thrown to position t. The fixed resistances A and B are equal, so that if $T = D$ the bridge would be balanced and the galvanometer would show no deflection. The resistance of T is, however, made considerably greater than that of D, so that the bridge is out of balance. By varying the resistance VR, the pointer of the galvanometer may be adjusted to some definite mark on the scale. This preliminary setting fixes the value of the current flowing in the main circuit due to the battery E.

For a temperature measurement the switch S is thrown to position 1. The couple and the resistances $X+C$ now constitute an arm of the bridge. The resistance C has a high temperature coefficient and is located at the cold junction of the couple. Suppose the apparatus is standardized for a cold-junction temperature of 0° C. Then, at this temperature $X+C=D$, and the galvanometer shows no deflection when the temperature of the hot junction is 0° C. As the temperature of the hot junction increases the emf developed by the couple increases and the bridge is thrown out of balance, causing a deflection of the galvanometer. The scale of the instrument is accordingly empirically graduated to read the temperature of the hot junction when the cold junction is at 0° C, and when the proper current flows through the main battery circuit as determined by the preliminary adjustment. When the temperature of the cold junction increases, the emf of the couple decreases, but the resistance C increases. An increase in the resistance C tends to increase the deflection of the galvanometer, while a decrease in the emf of the couple tends to decrease the deflection. On account of the fact that the thermoelectric power of a couple and the temperature coefficient of resistance of the coil C are nearly constant or vary similarly with temperature over a small range of temperature, by properly proportioning the various resistances of the circuit the increase in deflection due to an increase in the resistance of C compensates to all practical purposes for the decrease in deflection due to the decrease in emf of the couple as the temperature of the cold junction increases.

(*f*) Methods Employed in Correcting for Variations in the Temperature-emf Relation of Different Couples of the Same Type.—Platinum and platinum-rhodium suitable for thermocouples are refined by Engelhard in this country and by Johnson-Matthey in England. The temperature-emf relations for the couples obtained from these two sources differ from each other somewhat, but as stated elsewhere, the reproducibility of either of these general types of Le Chatelier couple is highly satisfactory. Base-metal couples of any given type, however, may produce under the same temperature conditions emfs differing by 0 to 5 per cent or more. Expressed in temperature, these differences may amount to 50° C at 1000° C. If the manufacturer of the couples exercises special care in the choice of the wire, these differences may be considerably reduced. For example, a certain

length of chromel wire and a length of alumel wire may be selected as representative of the standard couple for which the scale of the pyrometer indicator is graduated. The various stock coils of alumel are tested thermoelectrically against the standard alumel wire, and the stock coils of chromel against the standard chromel wire. If no emf is developed by heating the junction of the standard wire and the wire under test, it indicates that the two are similar. Suppose, however, that an emf of 0.5 millivolt is observed, the standard alumel wire being positive. This coil of alumel wire should be used accordingly with a coil of chromel wire to which the standard chromel wire tested 0.5 millivolt positive. By carrying through a series of such tests on many coils of wire, always heating to the same temperature, various pairs of chromel and alumel coils may be selected so that their temperature-emf relations are nearly the same as that of the standard couple. When these pairs of coils are made into couples, any differences in calibrations from that of the standard couple will be due mainly to heterogeneity of the wire itself. Usually a coil of wire is drawn from a single ingot, and the variations in the thermoelectric properties through a single ingot are likely to be far less than the differences in two ingots which may come from different melts.

After taking these precautions in regard to the selection of the wire, small variations in the emf-temperature relation of different couples still exist. Usually, they are of little practical importance, and most manufacturers consider couples made from wire thus selected to be sufficiently reproducible for industrial purposes. The difficulty in further correcting for the variations of individual couples from the manufacturing standpoint will be readily apparent.

(1) *Compensation by Series Resistance.*—It is entirely impractical to graduate the scale of the indicator for an individual couple because every time the couple is renewed a new scale would be required, and frequently many couples are used at the same time with a single indicator. If a couple shows higher emf than the standard or normal couple for which the indicator is graduated, an amount of resistance necessary to reduce the deflection to the proper value may be placed in series with the galvanometer. Usually, if a base-metal couple shows an emf, say 2 per cent high at 1000°C, it will be 2 per cent high at all other temperatures; so that this method of correction is satisfactory for all temperature ranges. The series resistance must be located at the couple and

not inside the galvanometer, for in the latter case compensation would apply for one couple only. If a couple shows emf 2 per cent high, the extra series resistance must be 2 per cent of the total resistance of the circuit. Thus, with a 300-ohm galvanometer and negligible line resistance, 6 ohms of magnanin is placed in series with the couple. This is mounted on a spool inside the terminal head of the couple. The use of a comparatively high *constant* line resistance is no serious disadvantage when an instrument is calibrated to read correctly for this resistance. The same galvanometer may be used with different couples; for example, one showing emf 2 per cent high, another 1 per cent high, and another 0.5 per cent high. When the resistances mounted in the heads of these couples have the values 6 ohms, 3 ohms, and 1.5 ohms, respectively, the temperature scale of the galvanometer is correct for all three. If the emf of a couple is low, resistance must be taken out of the circuit. The scale of the instrument is accordingly designed for the normal couple and for a certain normal series resistance, the latter being sufficient to permit adjustment for couples showing low emf. In the above example 6 ohms could be removed from the indicator and mounted in the head of the normal couple. This is the normal series resistance. For couples showing emf 2 per cent, 1 per cent, and 0.5 per cent high, the series resistances are 12, 9, and 7.5 ohms, respectively. For couples showing emf 2 per cent, 1 per cent, and 0.5 per cent low the series resistances are 0, 3, and 4.5 ohms, respectively.

This method of compensation is open to objection, however, especially from the standpoint of the pyrometer manufacturer, in that the resistance of the series coil required for exact compensation depends upon the resistance of the indicator. The following table shows the values of the series resistances required for indicators of various resistances in order to take care of a maximum variation of ± 2 per cent in the emf of different couples. The resistance of the line proper—that is, lead wires and couple without series coil—is assumed negligible.

Now, the development of the past five years in pyrometry has tended toward the production of indicators of higher resistance with the object of minimizing the errors arising from variations in the line resistance. The result of this development has placed on the market during this interval, in the case of a single manufacturer, instruments having resistances anywhere from 5 to 600 ohms and even 1200 ohms. Hence, in ordering couples for replace-

ment, the customer must state the resistance of the indicator and the manufacturer must carry in stock an almost endless assortment of couples compensated and calibrated to fit all the instruments he has manufactured possibly in the past 10 years. A large plant which has purchased instruments during an interval of five years may have an assortment of say 100 indicators with resistances from 50 to 600 ohms, all calibrated to read correctly for the normal couple. Since the compensated renewing couples are no longer interchangeable, this plant would be required to carry a stock of couples for every indicator. The chances for confusion of records and mixing of couples and the extra cost of such a complete stock are serious items.

TABLE 12.—Compensation by Series Resistance

Resistance of indicator	500 ohms	250 ohms	125 ohms	50 ohms	25 ohms	10 ohms
Emf of couple	Resistance of series coil for compensation					
	Ohms	Ohms	Ohms	Ohms	Ohms	Ohms
2 per cent low	0	0	0	0	0	0
1 per cent low	5.0	2.4	1.2	0.50	0.24	0.10
0.5 per cent low	7.5	3.7	1.9	.75	.37	.15
Normal	10.1	5.0	2.5	1.01	.50	.20
0.5 per cent high	12.7	6.3	3.1	1.27	.63	.25
1 per cent high	15.2	7.6	3.8	1.52	.76	.30
2 per cent high	20.3	10.1	5.0	2.03	1.01	.40

The error which is possible when a compensated couple is used with the wrong indicating instrument is illustrated by the following example: Suppose the couple originally read 2 per cent high and that it is compensated to read correctly with a 500-ohm indicator. In the plant the couple, by mistake, is connected to a 50-ohm instrument. The series resistance for the 500-ohm instrument is 20.3 ohms (see Table 12), while that for the 50-ohm indicator is only 2.03 ohms. The introduction of 20.3 ohms in series with a 50-ohm galvanometer when only 2.03 ohms in series should be used makes the instrument read 26 per cent low. Hence if the temperature of the furnace were 1000° C, the 50-ohm indicator would read about 740° C, or in error by 260° C. If no compensation whatever had been employed, and if the instruments were graduated for the normal couple with no resistance in series, each instrument when connected to the couple showing emf 2 per cent high would read 2 per cent high, or in error by about 20° C at 1000° C. Thus the error developed by an interchanging of instruments with the

compensated couple, a mistake which will occur frequently in any large plant, may cause an error many times as great as the error arising from the worst case of irreproducibility of couples when no compensating device at all is employed. A remedy for the prevention of such mistakes is to use instruments of one resistance only. Pyrometer indicators having a practically fixed preassigned resistance are made by certain manufacturers. All parts of the galvanometers are constructed according to specifications rigidly adhered to. The swamping resistance is finally adjusted until the total resistance of the instrument has the preassigned value found by experiment to be satisfactory. Slight differences will then exist in the calibration of different instruments. These can be corrected for by any of several methods such as illustrated below: (*a*) Calibrate the instrument by direct experiment and make a hand-drawn scale. Different instruments will have slightly different temperature ranges. (*b*) Use stock printed scales and adjust the sensitivity of the galvanometer by means of a magnetic shunt. (*c*) Use stock printed scales in several different temperature ranges and select the one which best fits the instrument. (*d*) Shunt the moving coil to give a specified deflection on a specified current. Then adjust the external resistance until the total resistance has the proper value. Use stock printed scales.

The adoption of a fixed standard instrument is, however, likely to thwart development work on the part of the manufacturers. Furthermore, instruments of all resistances and scale ranges are in daily use in the industries, and they are giving satisfactory service. It is impossible to consider the discarding of all indicators except those having a certain definite resistance in order that a convenient method of compensation by series resistance may be adopted. The objection to compensation by series resistance is further emphasized when indicators or an indicator and a recorder are operated in parallel on the same couple. Suppose a couple reading normally 2 per cent high is installed with a 500-ohm indicator. The series resistance in the head of the couple, as seen from Table 12, is 20.3 ohms. It is desired to operate another similar indicator in parallel, making two indicators of the same type on the same couple. The resistance of two 500-ohm indicators in parallel is 250 ohms. The two indicators accordingly act as a single indicator having a resistance of 250 ohms. Hence the series resistance must be 10.1 ohms instead of 20.3 ohms. If the couple is to be used with both indicators, it can never be used with the indicators

separately. If a 500-ohm indicator and a 125-ohm recorder are operated in parallel, the two instruments act as a single indicator, having a resistance of 100 ohms. For the above couple reading 2 per cent high the series resistance must be 20.3 ohms when the 500-ohm indicator is used, 5 ohms when the 125-ohm recorder is used, and about 4 ohms when both are used in parallel. In purchasing such compensated couples it is accordingly necessary to specify the resistances of the indicators and recorders, and to state the method of use—that is, to give a wiring diagram of the installation—and then the couple must be used always in the manner specified. When instruments of very low and different resistances are operated in parallel, the problem of choosing the proper compensation for the couple becomes very complicated. In fact, it is frequently necessary to use cut-out switches, so that the recorder is thrown out of the circuit when the indicator is read, and vice versa. A final objection to the method of compensation by series resistance is that it has no effect whatever when a potentiometric or semipotentiometric indicator or recorder is employed. A couple showing emf 2 per cent high and compensated to read correctly on a 500-ohm indicator will still read 2 per cent high when the measurements are made with a potentiometer. Since the use of potentiometric instruments is becoming more extensive every year, especially for laboratory and checking work, and for recorders, this additional objection to the method of compensation by series resistance is worthy of consideration.

(2) *Compensation by Shunt Resistance.*—In this method of compensation the thermocouple is shunted usually by a small resistance. If the couple normally reads high, the resistance of the shunt is decreased; if low, the resistance is increased. An ordinary base-metal couple for industrial purposes has a resistance of the order of 0.1 to 0.3 ohm at room temperature. Suppose this couple is shunted by a resistance of about the same magnitude. Such a shunted couple may be used with a potentiometer or with a galvanometer having almost any resistance from, say, 10 ohms up.

Various couples are thus perfectly interchangeable and may be used with instruments in parallel when desired. Hence none of the objections to the method of compensation by series resistance apply to the shunted couple. A very serious objection, however, may be raised against the method of shunted resistance in that the resistance of the shunt for proper compensation depends upon the resistance of the couple. The latter is subject to change with

temperature of the furnace and depth of immersion, depending upon the temperature coefficient of resistance of the two alloys forming the couple, and the resistance is, of course, altered by deterioration of the couple with use. The following example illustrates the error which may be expected from a slight change in resistance of the couple when the resistance of the shunt is low.

Suppose the couple having a resistance at room temperature of 0.2 ohm is shunted by 0.2 ohm of manganin. If e is the emf developed by the couple, the potential drop over the shunt is $e/2$ The scale of the galvanometer is arbitrarily graduated to take account of this reduction of emf. Let the resistance of the couple change from 0.2 to 0.25 ohm. This small change may be due to oxidation or may be accounted for by immersing the couple to half its length in a furnace at about 600° C. The potential drop across the shunt of 0.2 ohm on a couple of 0.25 ohm is $0.44e$ instead of $0.5e$, for which the galvanometer is graduated. The galvanometer accordingly reads 12 per cent low or in error by about 120° C at 1000° C. This error is many times greater than any error which would be introduced on account of irreproducibility of the couples if no compensation device whatever were employed. Hence, in attempting to correct for a small error of, say, 15 or 30° C, we have employed a method which is very likely to develop errors amounting to 100° C or more. The use of a shunt of low resistance has been discontinued. The method of shunted resistance is satisfactory, however, when the shunt has a resistance many times that of the couple. As the resistance of the shunt is increased the range for adjustment of different couples is decreased. However, it is not difficult to secure matched wire which is thermoelectrically reproducible to ± 2 per cent, and this variation can be taken care of by means of shunts of fairly high resistance. For generality the method is discussed under the following heading:

(3) *Compensation by Shunt and Series Resistance* [13].—Let r_1 = resistance of the couple + a small resistance (if necessary) in series with the couple; r_2 = resistance of the shunt; and R = resistance of the galvanometer. The potential drop E' across the shunt is given by the following equation where e is the emf developed by the couple at any given temperature; for example, 900° C:

$$E' = \frac{r_2 e}{r_1 + r_2 + r_1 r_2 / R} \qquad (1)$$

[13] Zimmerschied, U. S. Letters Patent No. 776252; 1915.

In case the potential drop is measured by a potentiometer we have:

$$E = \frac{r_2 e}{r_1 + r_2}. \qquad (2)$$

The shunt resistance r_2 is adjusted to compensate for the variation in emf of the different couples. On account of the term $\frac{r_1 r_2}{R}$ in equation (1) galvanometers having different resistances in general will be differently affected by variations in r_2 from couple to couple. If, however, the term $\frac{r_1 r_2}{R}$ is small enough compared with $(r_1 + r_2)$ this effect is negligible, and the potential drop across r_2 will be practically the same for all values of R and for a potentiometer, regardless of the values of r_2 for different couples. Let us impose the condition that the value of r_1, r_2, and R must be such that the reading with a potentiometer can never differ from that with a galvanometer by more than 0.5 per cent (that is, 5° C at 1000° C). In general, this difference will be found to be very much less. This condition is expressed by equation (3):

$$\frac{r_1 r_2}{R} \leqslant 0.005(r_1 + r_2) \qquad (3)$$

We desire to compensate for couples showing emfs differing from the normal couple by $< \pm 2$ per cent. For convenience in making the adjustments on the shunt it is better to allow a little more variation for couples showing low emf. We will make the computations so that a couple reading 3 per cent low could be compensated for by using a shunt of infinite resistance—that is, with no shunt at all. If e is the emf of any couple, at some fixed temperature, and e' is the emf of a couple 3 per cent below normal, we have from equation (2) for compensation:

$$\frac{r_2 e}{r_1 + r_2} = \frac{r'_2 e'}{r_1 + r'_2} = e', \text{ since } r'_2 = \infty. \qquad (4)$$

Hence, substituting in (3) the value of r_2 found from (4),

$$r_1 \leqslant \frac{R}{200}\frac{e}{e'} = \frac{R}{200} \cdot \frac{\text{emf of any couple}}{\text{emf of couple 3 per cent low}}. \qquad (5)$$

It is of advantage to make r_1 as large as possible. This can be done by making R large, but the value of R must be small enough to take care of all galvanometers likely to be employed. If we denote by R_0 the lowest galvanometer resistance for which com-

pensation is required, we have equation (6) for the maximum desirable value for the resistance of the couple.

$$r_1 = \frac{R_0}{200} \cdot \frac{e}{e'} = \text{total resistance of couple.} \qquad (6)$$

Substituting this value of r_1 in equation (4) we obtain:

$$r_2 = \frac{r_1}{\left(\frac{e}{e'} - 1\right)} = \frac{R_0}{200\left(1 - \frac{e'}{e}\right)} = \text{shunt resistance.} \qquad (7)$$

Table 13 shows the values of the shunt resistances and couple resistances for the minimum galvanometer resistances 100, 80, 60, and 40 ohms. The compensation is better the more the resistance of the galvanometer exceeds these minimum values, and in no case does the error in compensation amount to more than 0.5 per cent (that is, 3° at 600° C or 5° at 1000° C).

TABLE 13.—Compensation by Shunt and Series Resistance

Series resistance	r_1=0.5 ohm	r_1=0.4 ohm	r_1=0.3 ohm	r_1=0.2 ohm
Couple	Shunt	Shunt	Shunt	Shunt
	Ohms	Ohms	Ohms	Ohms
3 per cent low	∞	∞	∞	∞
2 per cent low	48.50	38.80	29.10	19.40
1 per cent low	24.25	19.40	14.55	9.70
Normal	16.17	12.94	9.70	6.47
1 per cent high	12.12	9.70	7.27	4.85
2 per cent high	9.70	7.76	5.82	3.88
Minimum galvanometer resistance	100	80	60	40

In using this method of compensation the standard galvanometer scale is graduated in the usual manner for the couple which reads 3 per cent low. Thus, if we have a table of emf versus temperature for the normal couple, we decrease all the values of the emfs in the table by 3 per cent. If 800° C corresponds to an emf of 33.2 millivolts for the normal couple, the 800° C point on the galvanometer scale must be equivalent to (0.97)(33.2), or 32.2 millivolts, and, similarly, for all the other graduations.

It was noted in the case of shunting a couple of 0.2 by 0.2 ohm, that if the resistance of the couple increased from 0.20 to 0.25 ohm due to change in depth of immersion, the galvanometer would be in error by 120° at 1000° C. On referring to Table 13 the greatest error which a change in the couple resistance from 0.20 to 0.25 ohm can produce when properly shunted occurs with the couple

reading normally 2 per cent high, in which case the shunt has a resistance of 3.88 ohms. The error due to this change in resistance amounts to 1.2 per cent, or about 12° at 1000° C instead of 120° C. For the normal couple the error is only 7° C and for the couple reading 2 per cent low only 3° C. The error thus decreases as the resistance of the shunt increases. This shows that it is more desirable to use a couple having a resistance of 0.5 ohm. If the resistance of the couple alone is only 0.2 ohm, 0.3 ohm of manganin may be used in series, and the shunt is connected over the total of 0.5 ohm. If the resistance of the couple is now altered by 0.05 ohm from 0.50 to 0.55 ohm the couple reading 2 per cent high, normal, and 2 per cent low will be in error by only 5, 3, and 1° C at 1000° C, respectively. It is evident that this method of compensation is far superior to the use of a shunt of low resistance.

(4) *Summary Statement on Reproducibility of Couples.*—The object of this section has been primarily to call the attention of the pyrometer engineers in the industrial plants to the difficulties encountered by the pyrometer manufacturer, when he attempts to correct for small variations in the calibration of different couples. The Bureau of Standards has calibrated chromel-alumel couples submitted for test which deviated from the normal couple by 20° C at 1000° C. On the other hand, the Bureau has purchased chromel-alumel wire at different times for which the maximum deviation from the specified temperature-emf relation was only 4° C. It is certainly possible, if necessary, to hold the manufacturer to within ±5° C of the specifications for an uncompensated couple. There are few industrial processes, however, using base-metal couples which require an accuracy of even 20° C at 1000° C, and there are still fewer processes in which temperatures are measured to this accuracy or would be likely to be measured to this accuracy even if a perfectly compensated couple were secured. The users of pyrometers have forced these compensation methods upon the manufacturer by insisting upon greater precision than is really necessary. In so doing the user obtains a couple which when installed in a certain precise manner and frequently checked may give satisfactory results; but usually the compensation device is a source of more serious error than would be occasioned by the slight irreproducibility of the uncompensated couples. In the past few years remarkable advances have been made in the precision required and attained by certain few industries in temperature regulation, notably in the heat treatment of high-grade steel, such as dies, etc. Here an accuracy

of 5° C is frequently necessary, and some manufacturers claim even greater precision than this in the reproducibility of the furnace conditions. Such precision, however, can not be expected of compensated couples. A solution of the problem from the manufacturing point of view is to secure as well matched wire as possible, do away with compensation devices, and sell at different prices two grades of couples, one which is guaranteed to ±5° C and the other to ±10° C. Possibly at a later time, closer specifications could be adopted. If any industrial process requires greater precision than this, the couples should be individually calibrated and correction curves prepared similar to those furnished with high-grade voltmeters and electrical instruments. If exact reproducibility and higher accuracy are both required, the rare-metal couple should be employed. In objecting to compensating methods, we refer only to those devices which are supposed to correct for variations in the thermoelectric characteristics of the couple wire. Compensation methods for eliminating cold-junction errors, ordinary "compensating lead wires," etc., are, of course, necessary, and these methods must not be confused with the methods discussed in the above section, which operate for an entirely different purpose.

(*g*) THERMOCOUPLE INSTALLATIONS—(1) *Special Precautions.*—The installation of a large thermocouple equipment requires the services of competent electricians. Just as much attention, if not more, should be given to the wiring, switches, switchboards, etc., as is given in the case of ordinary power installations. Proper fixtures should be used to mount the couple in the furnace. Lead wires should have a weatherproof covering and should be run in a metal conduit except for a short length of flexible cable at the ends of the conduit. The conduit should be grounded to prevent leakage from power installations or lighting circuits. All joints in the lead wires should be soldered and taped. When indicators or recorders of low resistance are employed, it is of the greatest importance to have a well-constructed electrical installation to insure a constant line resistance. Since instruments of low resistance are usually calibrated for a low line resistance of definite value, the size of copper wire required for a long line may be as large as No. 12 or No. 10. Special attention must be given to contact resistances at switches. Frequently switches rated at 100 amperes are required, although the actual thermoelectric current is only a few milliamperes. If the indicator is of high resistance, or is equipped with a compensating device, or operates

upon the potentiometric or semipotentiometric principle, the factor of very low line resistance is not of great importance, but the wiring should be well installed for the psychological effect, if nothing else. Stationary indicating and recording instruments usually should be mounted upon switchboards, with suitable selective or commutating switches when several couples are used with one indicator. When the head of the couple is exposed to severe conditions, rain, etc., as in outside kilns, a weatherproof terminal head should be used. This consists of an outside casing which fits over both binding posts. The cover may be tapped for conduit wiring or provided with a packing gland or stuffing box if a length of flexible cable is used between the couple and the conduit. The lead wires should be carried from the couple to the indicator through as cool rooms as conveniently possible. Copper has a high temperature coefficient of resistance, and the frequent practice of running wires over the top of a long row of furnaces may cause large variations in line resistance.

The indicator or recorder should be conveniently located. If the instrument is desired especially for the use of the operator of a furnace, it should be placed where it is readily available. It should be mounted where vibration and excessive dirt and dust will not injure the delicate part of the mechanism. In almost all industrial installations outside protecting cases are required to prevent dust from filtering through the case of the indicator. A suitable protecting case is illustrated by Fig. 38. The various screws and gears of a recorder require occasional attention. The tools for this work, screw drivers, oil can, etc., may be mounted inside the outer protecting case, where they are available for immediate use. Special devices are employed to damp out vibration when this is serious, as in the neighborhood of a trip hammer or rolling mill. Frequently the instruments are suspended on spiral springs. One convenient method especially suitable for heavy instruments, such as a recorder, is to mount the instrument on a shelf which is supported on a pier by four ordinary tennis balls, one at each corner of the shelf. The tennis balls damp out vibration very satisfactorily.

The proper location of couples in the furnace depends upon the particular process and use to which the furnace is put. The primary consideration must be to locate the hot junction at the point the temperature of which is required. A secondary consideration, however, is to locate the couple where the lead wires may

be conveniently taken care of. The space between the protecting tube of the couple and the furnace wall should be tightly plugged with refractory cement, so that hot air can not strike through the hole onto the head of the couple, nor cold air be drawn in, thus cooling the hot junction of the couple.

The use of extension or compensating lead wires, cold-junction boxes, etc., has been discussed elsewhere. The cold-junction box should be so located as to reduce the amount of compensating lead wire required to a minimum. Compensating lead wires, except ordinary iron-constantan leads, are somewhat costly and should not be employed extravagantly. Also the use of long

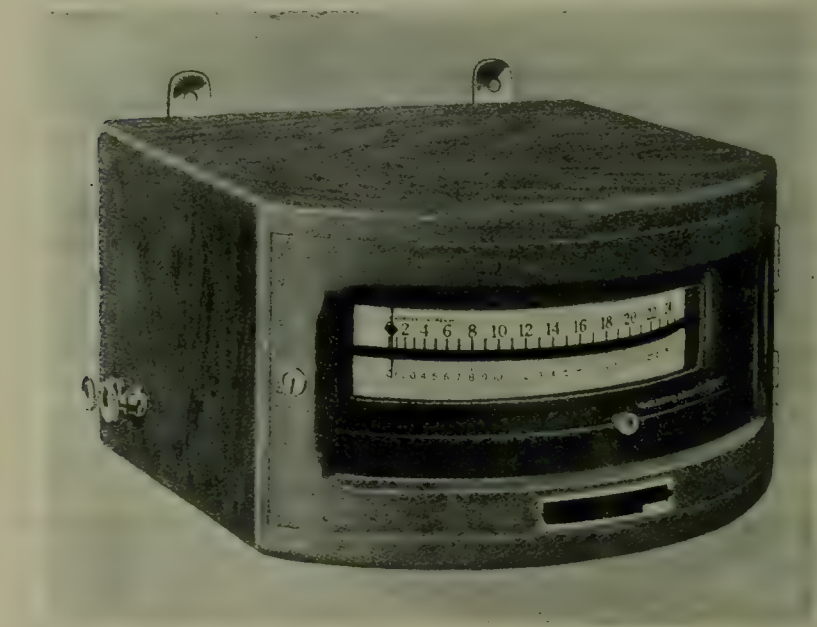


FIG. 38.—*Indicator in hardwood protecting case*

lengths of compensating wire increases the line resistance, since the resistivity of these leads is much higher than that of copper.

In case the cold junction is buried under ground, it must not be located too near a large furnace. Either the distance from the furnace or the depth at which the junction is buried must be increased. A depth of 10 feet at at least 10 feet from a large furnace is usually satisfactory.

(2) *Common Return.*—The use of a common-return wire for a multiple installation is in general unsatisfactory. With such installations, short circuits through the metal protecting tube of

the couple to the furnace and other couples are likely to occur. The trouble which the short circuits can produce is sufficient to warrant the extra cost of copper required to prevent them. With the common return, leakage from a power installation affects the reading of every couple connected to the return and a leakage through a high resistance may alter the readings of every couple by the same amount so that the presence of such leaks is not always readily detected. It is also possible by leakage from different couples to the ground to obtain very erratic and erroneous readings when the common return is employed. Base-metal couples are frequently constructed with the hot junction welded to the end of the iron protecting tube in order to reduce thermal lag. Even when this welded junction is not made, the hot junction usually touches the protecting tube and is in good electrical contact with it, especially since at high temperatures insulation resistance becomes very low. Suppose that the iron tubes of two chromel-alumel couples are grounded to the iron casing of a furnace. The two hot junctions are thus connected to each other by a circuit of iron. The electrical circuit is represented by Fig. 39. On the indicator we have a chromel-alumel couple, one leg of which is shunted by an alumel-iron-alumel differential couple. If the temperatures of the hot junctions of the couples are the same, this differential couple produces no effect. It will, however, alter the reading of the indicator whenever the two temperatures differ. Both the chromel-alumel couples will accordingly give erroneous results. If individual returns are used, the iron circuit produces no effect. When grounds occur farther back from the hot junction—for example, between the common return and the other lead wire of a single couple—all couples on the common return have, in addition to their own emf, an impressed potential drop due to the current flowing in the shorted couple. This may cause a large error in the reading of every couple on the line. Installations employing a common return are extensively used in the

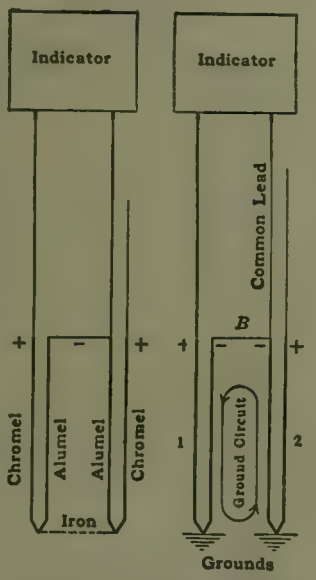


FIG. 39.—*Illustrating objection to common return*

industries. It is an unwise practice, and one which should be avoided as far as possible.

(3) *Wiring Diagrams of Thermocouple Installations.*—Fig. 40 illustrates a simple thermoelectric installation for a rare-metal couple. The couple is properly protected by a porcelain or quartz tube and, if necessary, by an outer tube of iron, chromel, fire clay, etc. From the head of the couple compensating lead wires are carried to the bottom of a pipe driven 10 feet under ground. From the bottom of the pipe copper lead wires are carried to the indicator.

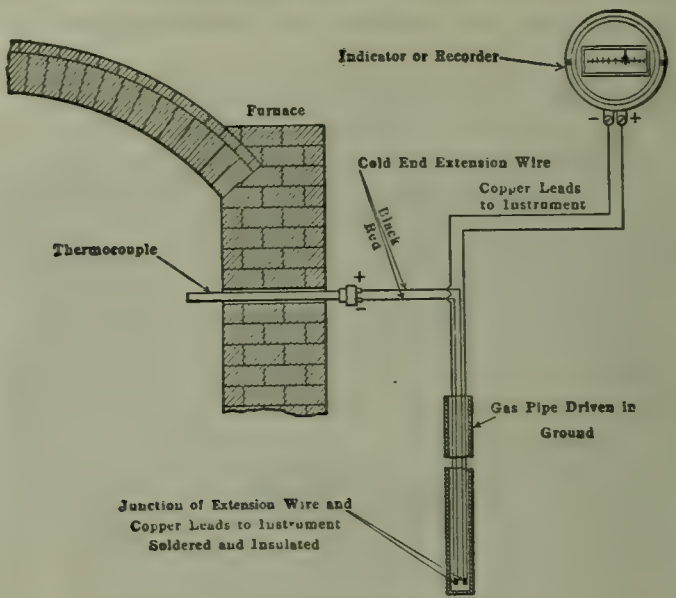


FIG. 40.—*A simple thermocouple installation*

Fig. 41 illustrates a multiple installation for five thermocouples. In this case a common return is employed, although, as stated above, the use of an individual return is preferred. By use of the common return for this installation four lengths of copper wire (or compensating leads) from the couples to the recorder have been saved, and the commutating switch is simpler. The indicator for the operator of the furnaces and the recorder for the superintendent's office are mounted in parallel. The indicator or recorder may be connected to any couple desired by setting the commutating switch illustrated in the lower halves of the cases of the instruments. Such an installation can be employed only when the instruments have a high resistance. The recorder and indicator, when connected to the same couple at the same time, act as

a shunt on each other, and this tends to make both instruments read low, whereas, if the two instruments are calibrated to read correctly in parallel, they will both read high when connected to different couples. An example illustrates this point. Suppose the line and couple resistance for each circuit is 3 ohms and the resistances of the recorder and indicator are 500 ohms each. Assume that both instruments are calibrated to read correctly when connected separately to any couple. The potential drop E across the terminals of either instrument bears the following relation to e, the emf of the couple:

$$E = \frac{500}{503} e.$$

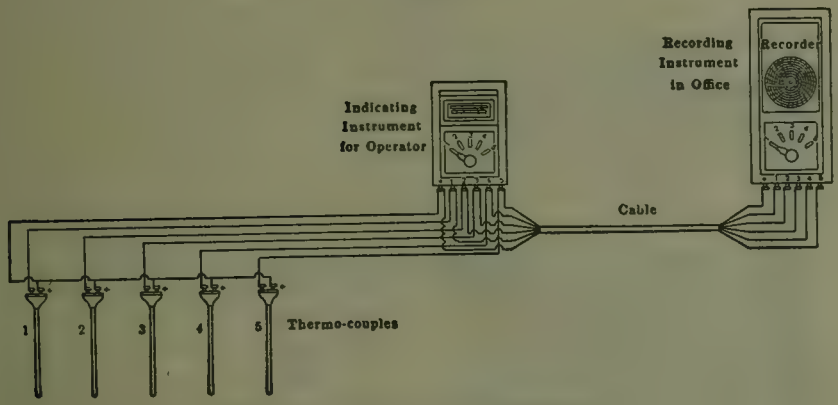


FIG. 41.—*A simple 5-couple installation*

The scale of the instrument is graduated to take account of this reduction in emf. When the two instruments are connected in parallel, the potential drop across the indicator and recorder is given by the following equation, where R equals resistance of indicator or recorder and r equals line resistance:

$$E' = \frac{Re}{R + 2r} = \frac{500}{506} e.$$

There is, accordingly, a reduction in emf by the factor $\frac{500}{506}$, when the instruments are connected in parallel to the same couple. Each instrument is calibrated for a reduction in emf of $\frac{500}{503}$. The error resulting from the parallel connection is thus 0.6 per cent, or about 6° C at 1000° C. Hence, if the operator of the furnace switches couple number 5, for example, on the indicator when this couple also is connected to the recorder, both instru-

ments will read about 6° C low at 1000° C. This error is usually insignificant.

A similar example will be considered for an indicator and recorder of low resistance. Let the line resistance r equal 3 ohms as before, and the resistances R of the indicator and of the recorder equal 10 ohms each. The potential drop E across the terminals of either instrument bears the following relation to e, the emf of the couple:

$$E = \frac{Re}{R+r} = \frac{10}{13}e.$$

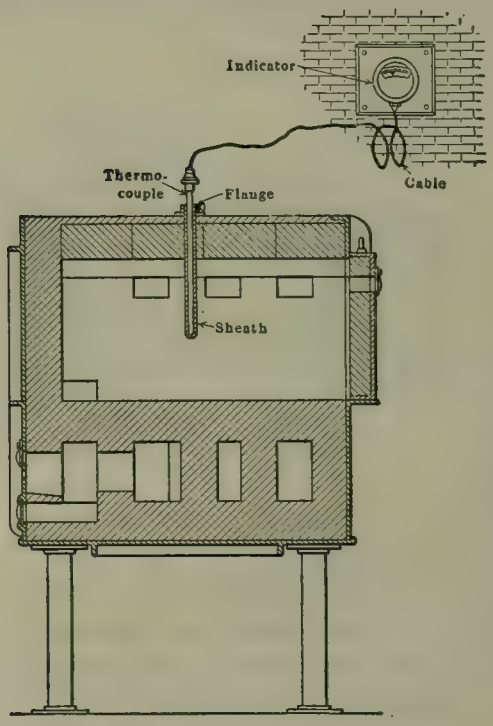


FIG. 42.—*Thermocouple in oil-fired furnace*

When the two instruments are connected in parallel, the potential drop across the indicator and recorder is as follows:

$$E' = \frac{R}{R+2r}e = \frac{10}{16}e.$$

The instruments are calibrated to read correctly when used separately; that is, for a reduction in emf by the factor 10/13. When used in parallel there is a reduction of emf by the factor 10/16. The error resulting from a parallel connection is thus 19 per cent or about 190° C at 1000° C. Hence, if the operator of the furnace switches couple number 5, for example, on the indicator when this couple is connected to the recorder, both instruments will read low by 190° C at 1000° C. This is a very serious error. Accordingly, instruments of low resistance can not be operated alternately separately and in parallel on the same couple. They must be used *always* either separately or in parallel. In either case the scales of the instruments are graduated for the proper reduction in emf due to line drop. The paralleling of simple galvanometric instruments having a resistance of 300 ohms and more, when the line resistance is less than 3 ohms, or of potentiometric instruments, is a safe practice. The paralleling of instruments of

lower resistance requires specially graduated scales or the use of special wiring circuits. Instruments of low resistance designed for parallel operation should not be used separately. Instruments of low resistance operated with cut-out switches may be used separately, since the switches are designed so, that while a recorder and indicator are operated on the same line, they are never connected to the same couple at the same time.

Fig. 42 illustrates a simple thermocouple installation in an oil-fired furnace. The thermocouple is mounted in an additional metal sheath to protect it from mechanical shocks and breakage. Compensating lead wire is carried to the indicator. The cold junction is located at the indicator and is not thermostatically

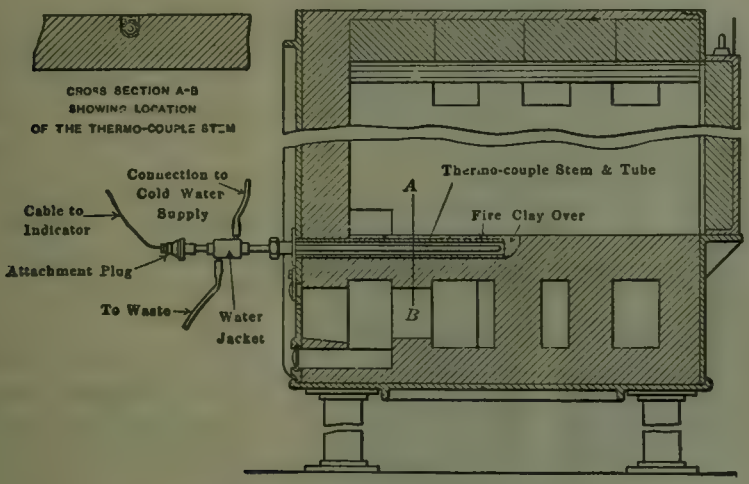


FIG. 43.—*Thermocouple in oil-fired furnace*

controlled. The indicator is set to read the room or cold-junction temperature on open circuit. Otherwise correction for "cold-junction error" must be applied.

Fig. 43 shows a thermocouple embedded in the floor of an oil-fired furnace. The couple thus requires no space in the heating chamber. The cold junction is water jacketed.

Fig. 44 shows a method of installing a couple in the wall of a large furnace.

Fig. 45 shows a method of installing a couple in a galvanizing tank or a pot of stereotype metal, babbitt, or tin.

Fig. 46 illustrates a multiple thermocouple installation connected to a single indicator. Compensating lead wires are carried

from the couples to a conveniently located cold-junction box. The temperature of this box is thermostatically controlled. From the cold-junction box copper wires are carried to the terminal block and selective switch illustrated. A common return has been employed between the cold-junction box and the switchboard. In general, it is preferable to use individual return wires for each couple. The switchboard illustrated is designed for six couples. By pressing one of the buttons shown any desired couple is connected directly to the indicator.

(4) *Commutating Switches.*—Fig. 47 shows a multiple rotary switch. By turning the dial to the proper position any one of 12 couples may be connected to the indicator. The commutating brushes in this switch are laminated phosphor-bronze, diagonal wiping, and have a long spring action to follow up all possible wear. Fig. 48 illustrates a switch of different design, but similar in principle. Double points of contact are required when individual return wires are employed. The positive wires of each couple are connected to the outer ring of contacts and the negative wires to the inner ring. The galvanometer is connected across the two solid rings. Commutating switches are designed so that variable contact resistance is reduced to a minimum.

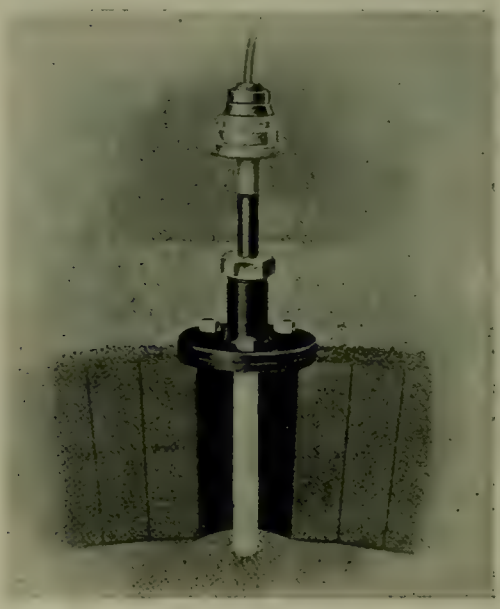


FIG. 44.—*Method of mounting couple in furnace*

For large installations several hundred couples may be connected to a switchboard, and the operator of the board connects the couples successively to the indicator. The switchboard is frequently designed somewhat similar to an ordinary telephone switchboard. Often in these large installations communication between the operator of the switchboard and the operator of the

furnace is maintained by a system of colored electric lamps. This method of temperature control is meeting with great success in the industries. A description of its applications is presented elsewhere.

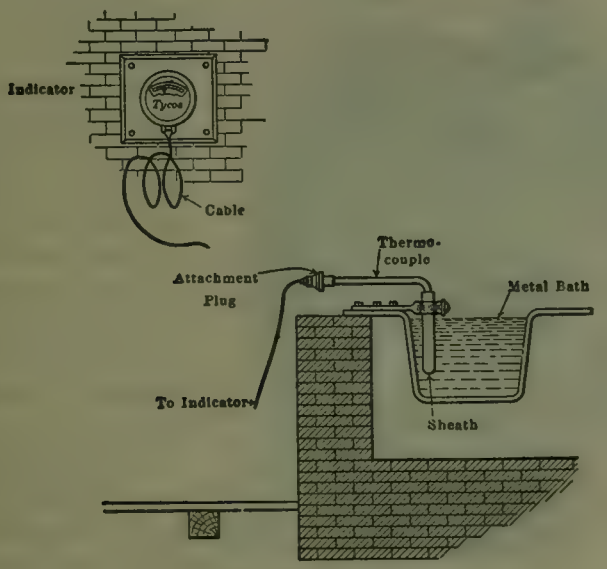


FIG. 45.—*Thermocouple in lead bath*

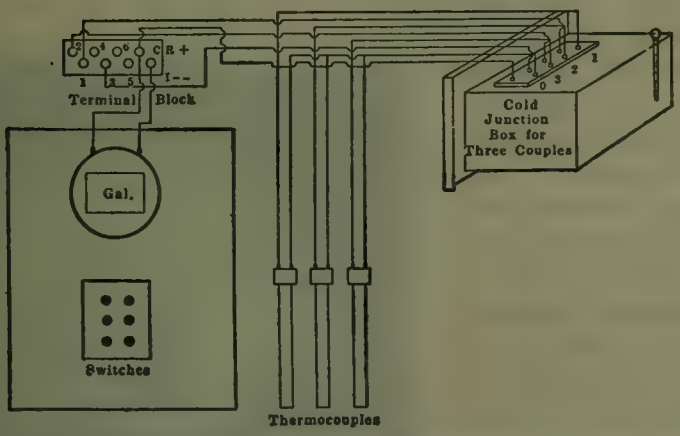


FIG. 46.—*Multiple-couple installation with thermostated cold-junction box and compensating leads*

(5) *Use of Junction Box.*—Fig. 49 illustrates a wiring diagram for a multiple-couple installation which is very useful in saving compensating lead wire and in thus reducing the cost and the resistance of the line. The junction box is a cast-iron box such as

those used for underground telephone wiring. The box is not thermostated, since a constant and measured temperature is not required. It is merely necessary to have a uniform temperature in the box. This is secured when the box is constructed of heavy metal. The emf developed at the junctions of compensating leads and copper leads is compensated for by a common junction in the opposite direction inserted between the selective switch and the indicator or recorder. A common cold junction is also placed here. In the illustration this is located in a pipe buried 10 feet under ground.

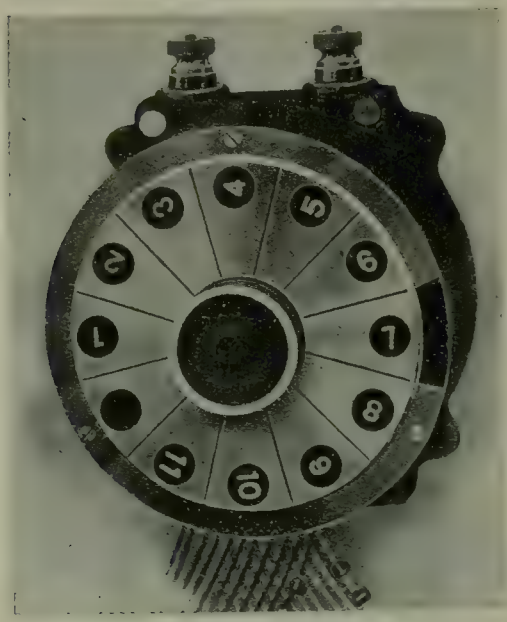


FIG. 47.—*Type of commutating switch*

The selective switch and recorder or indicator are usually mounted in a single case. The common cold junction and the junction-box compensating couple are connected at the recorder between the switch and the binding-post terminals of the instrument, as illustrated. The cold junction is placed near the junction box and the recorder or indicator (with switch, if desired) may be any distance away, since only copper leads are used from this point to the junction box. The method is especially useful where separate cold

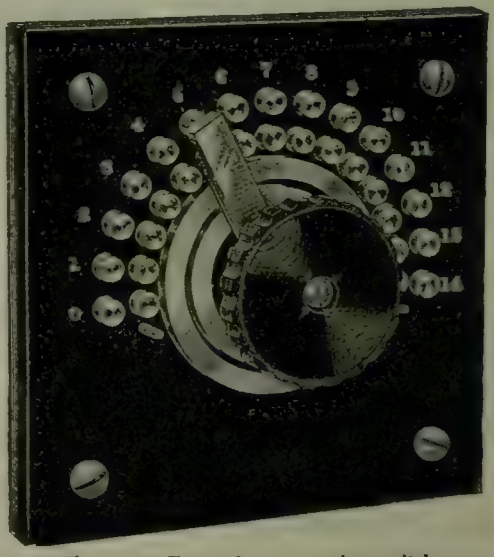


FIG. 48.—*Type of commutating switch*

junctions require too long compensating leads. The following example illustrates a case where such an installation is desirable. Suppose the temperature of a coke oven 20 by 40 by 150 feet is measured by nine couples inserted in the top. The indicator is located at the ground level 40 feet from the furnace. The buried cold junctions are 20 feet in front of the oven. The following illustrates the amount of compensated lead wire required to reach the buried cold junctions when the junction box is not employed: Three couples at rear of furnace $3(150+20+20+10)$ feet, three couples at center of furnace $3(75+20+20+10)$, three couples at front of furnace $3(20+20+10)$. Total compensating cable, 1,125 feet.

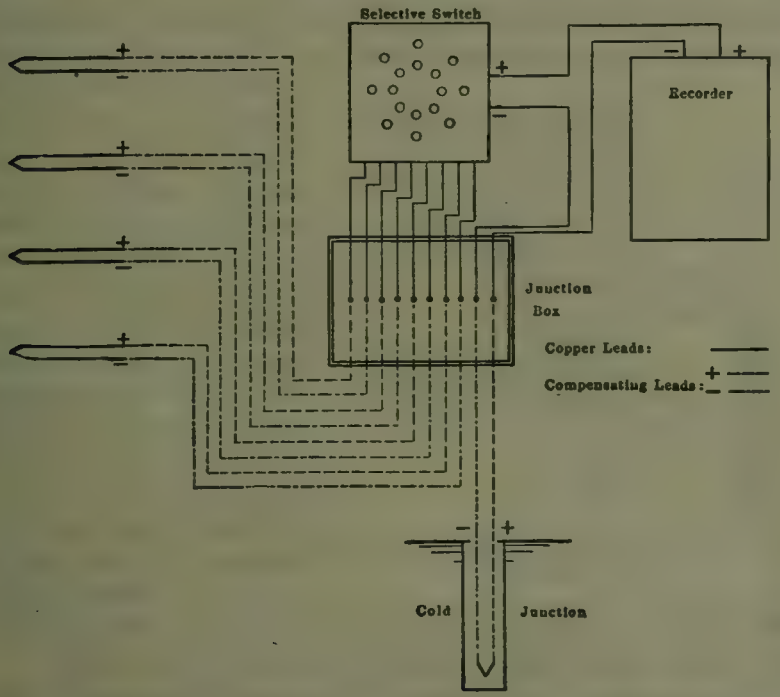


FIG. 49.—*Illustrating use of junction box with controlled cold-junction temperature*

Consider the same installation when a junction box is located on top of the furnace at the center: Three couples at rear of furnace $3(75)$ feet, three couples at center of furnace $2(10)$, three couples at front of furnace $3(75)$, from box to cold junction $(75+20+20+10)$. Total compensating cable, 595 feet.

By means of the junction box we effect a saving of some 500 feet in the compensating cable, and need to bury only one pair of junctions, and just as satisfactory an installation is obtained. In installing a large multiple-couple equipment with a junction box,

it is very important to insure that the common cold-junction couple is connected with the correct polarity as illustrated. Although we have used a common cold junction for all couples, we have not employed the objectionable common return. Individual returns are used with every couple shown in the diagram.

In case the recorder is placed where the temperature is quite uniform from day to day, the use of a buried cold junction or thermostated cold-junction box is not absolutely essential. The emf generated at the junction box in Fig. 49 is then compensated for by running one pair of compensating leads from the recorder to the junction box, taking care to connect the negative lead to the negative terminal of the recorder and the positive lead to the selective switch. A simple installation of this kind is illustrated by Fig. 50. Here only one couple is shown, but as many couples as desired may be connected to the multiple pole selective switch. The compensating lead wires are soldered together inside the junction box. The auxiliary couple formed by the compensating leads is in series with the couple connected in by the selective switch. The cold junction is accordingly at the recorder where the temperature is fairly constant. Changes in temperature of the distributing or junction box thus will not affect the reading of any couple.

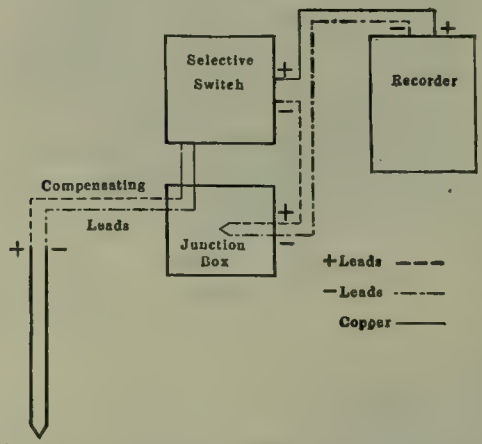


FIG. 50.—*Illustrating use of junction box without cold-junction control*

The principle of the junction box is somewhat similar to that of the Wilson-Maeulen zone box, except that no thermocouple in the junction-box system is connected directly to the compensating couple, all connections to the auxiliary couple being made through the selective switch. The couple $y\ z$ is connected directly to the zone box, Fig. 51. Two pairs of wires lead from the zone box, one pair of copper being connected to the main line and indicator. The other pair YZ are carried to the cold-junction box or are buried underground. For base-metal couples, Y and y are of the same material and also Z and z. For rare-metal couples Z and Y are copper and an alloy of copper-nickel, respec-

tively, the compensating leads already described for use with the Le Chatelier couple. The zone box thus saves running an extra pair of copper lead wires to the bottom of the cold-junction well. If this principle is used for a multiple installation with the object of saving compensating leads, a selective switch may be mounted between the zone box and the different couples. However, this requires that the selective switch be mounted near the furnace, unless a complicated interlacing electrical circuit is employed, whereas it is always desirable to have the switch at the indicator. It appears, therefore, that for large installations the junction box described above is preferable as far as economy in compensating lead wire is concerned. If a single-couple installation be used, or separate cold-junction wells for different couples in multiple installations, the zone box accomplishes practically the same results as the junction box, namely, a small saving in copper and frequently a more desirable wiring system. The main advantage, however, of the junction box is the saving of compensating lead wire in a multiple-couple installation. The main advantage of the zone box is the simple method by which the cold junction is extended to some point at which the temperature can be controlled or is constant; for example, the bottom of the cold-junction well.

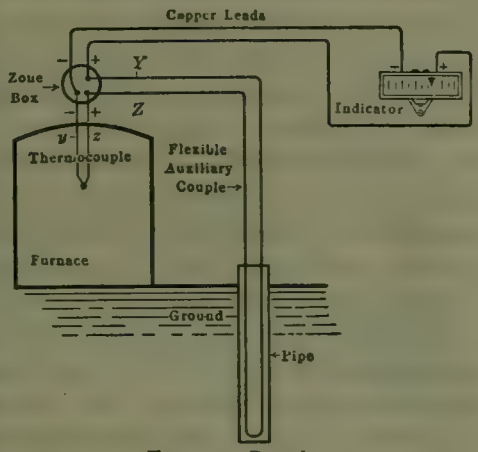


FIG. 51.—*Zone box*

(6) *Determination of Temperature of Buried Cold Junction.*—Several methods are available for obtaining the temperature at the bottom of the junction well. The simplest is to use a thermocouple consisting of the compensating leads. Insert this in the well, connect it to a portable indicator, and measure the secondary cold-junction temperature at the indicator with a thermometer. With a well 10 feet or more deep and properly located it is necessary to measure the temperature possibly only once a month. Another method is to lower a thermometer into the well. The thermometer is wrapped with a few layers of cloth, leaving the stem exposed near the expected reading. It is left in the well for thirty minutes and read quickly after raising. The recorder or indicator is

adjusted to correct for this temperature or the records may be marked "cold junction − °" and corrections applied later.

All buried leads to the cold junction should be rubber insulated, and the junction well should be made water-tight. The compensating leads, particularly those for base-metal couples, will generate a large voltaic emf if they are wet and not insulated with water-proof covering.

(7) *Depth of Immersion of Couples.*—Thermocouples immersed in furnaces of the various industrial types must be carefully protected. Heavy iron tubes and frequently larger auxiliary protecting tubes of various materials are employed. The thermocouple calibrated in the laboratory will give the temperature of the hot junction when installed in the protecting tubes, but there is no certainty that this temperature is that of the furnace. Conduction along the protecting tubes may be so great that the temperature of the couple is far below that of the furnace. Conduction losses affecting the temperature of the hot junction may be reduced and even eliminated by using a sufficient depth of immersion, but it is not always possible to do this, and it is difficult to determine when the depth of immersion is sufficient. For example, an immersion of 5 cm might be satisfactory for one type of furnace or for a molten bath, and 30 cm insufficient for a certain muffle furnace. Each particular case requires special consideration. The general practice is to use as deep immersion as is conveniently possible and trust that this is satisfactory. Two methods may be suggested for investigating the question, but neither one is very conclusive. First, remove the couple alone from the fixed installation, leaving all protecting tubes in place. If it is impossible to remove the couple from the iron tube, use a similar tube in the fixed installation without the couple. Explore the temperature inside the protecting tube with an unprotected couple. If the temperature for several centimeters along the axis of the tube at the inner end is practically uniform, the depth of immersion is satisfactory. If, however, the temperature falls rapidly in this region, the immersion is not deep enough. Second, the couple previously standardized is mounted complete in the fixed installation and compared with another checking couple mounted along the side of the permanently installed couple. The checking couple must have a small cross section and must be used either unprotected or protected by an extremely thin tube in order to minimize the loss of heat by conduction. The hot junctions of this couple and of

the couple under test are brought closely together, but not in contact. The checking couple should indicate a temperature more nearly that of the furnace than the temperature observed with the fixed couple. If the temperature difference between the two couples is large, a greater depth of immersion should be employed. These methods are complicated by local variations in the temperature of the furnace, but checks of this nature, although somewhat unsatisfactory, are better than no checks at all.

(8) *Couples Purposely Insufficiently Immersed.*—It is frequently desirable to purposely immerse the couple to an insufficient depth. In many processes the furnace is operated at such a high temperature that a thermocouple or protecting tube can not withstand the severe conditions to which it may be subjected. In this case the couple may be immersed only part way through the furnace wall, or to a distance flush with the inner wall of the furnace. The temperatures indicated by couples installed in this manner are always lower than those of the furnace interior, but they bear a fairly definite relation to the temperature of the furnace, and hence the method is satisfactory for temperature control and reproduction of furnace conditions from day to day.

(9) *Protecting Tubes for Thermocouples.*—The choice of a proper protection tube for a thermocouple is nearly as important as the selection of the material for the couple. Among others, the following properties of a protection tube should be considered in reference to the particular process for which the tube is required:

(*a*) Low Porosity to Gases.—Many tubes become very porous to furnace gases at high temperatures. Furnace gases usually attack the couple.

(*b*) Low Volatility.—Certain metal tubes are undesirable at high temperatures because the metal distills upon the couple, thus altering its calibration.

(*c*) Ability to withstand high temperature.

(*d*) Ability to withstand sudden changes in temperature.

(*e*) Ability to withstand mechanical shocks and strains.

(*f*) High Rigidity or Viscosity.—Protecting tubes frequently deform and exhibit the phenomenon of plastic flow at high temperatures.

(*g*) Thermal Conductivity.—High thermal conductivity is frequently desirable when rapidly changing temperatures are measured. Usually, however, low thermal conductivity is desired so that the flow of heat along the tube is as small as possible.

(*h*) Ability to resist corrosion from molten metals or furnace gases.

Excellent protecting tubes are obtainable for many different industrial processes. However, for certain processes satisfactory tubes have not as yet been developed. The problem is one for the ceramist or metallurgical engineer. This is particularly true in obtaining suitable tubes for molten metals, especially iron and brass. The following describes the more important protecting tubes now in use industrially:

Fused Quartz (Fig. 52, Nos. 1, 2, and 3).—No. 1 or smaller tubes are used as insulators through which the wires of a rare-metal couple are threaded before inserting into the protecting tubes 2 or 3. Tube 2 is of fused-quartz powder, which is afterwards

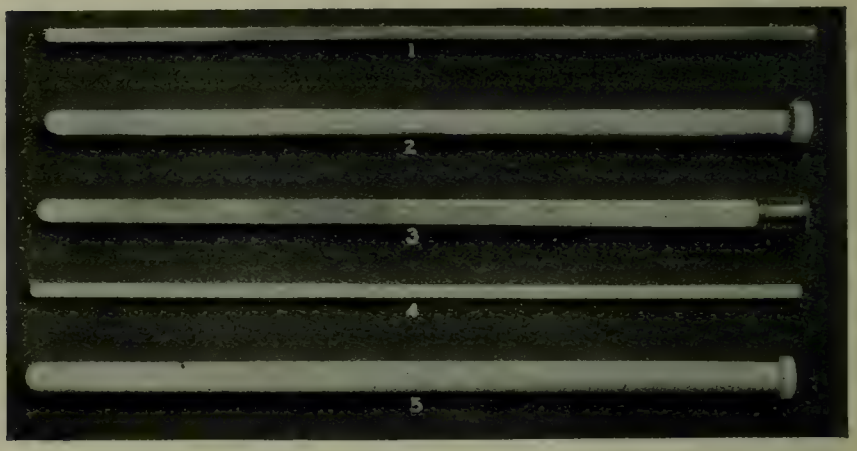


FIG. 52.—*Protecting tubes for couples*

fired. The preparation of such tubes is similar to that of porcelain tubes. Tubes 1 and 3 are drawn from molten quartz in much the same manner as in the manufacture of glass tubing. These tubes are translucent, like china. Transparent quartz-glass tubing is manufactured, but on account of its cost is not used industrially for thermocouple protection. Fused quartz affords good protection up to 1050° C in an oxidizing atmosphere free from alkalies. The material is somewhat pervious to hydrogen and probably to other reducing gases, but is not pervious at this temperature to oxygen or carbon dioxide. Any reducing gas within the protecting tube of a rare-metal couple is disastrous, particularly when the tube contains silica. The silica is reduced to silicon which is readily absorbed by platinum. Above 1050° C

and even at lower temperatures after prolonged heating, quartz devitrifies and crumbles away. Quartz tubes withstand sudden changes of temperature without breaking. Heavy molded quartz tubes are sometimes used for extra protection; for example, against acid fumes.

Porcelain (Fig. 52, Nos. 4 and 5).—Porcelain is used primarily for protection of rare-metal couples. Previous to 1914 a highly refractory porcelain known as Marquardt was imported from Germany. A better grade of this material was developed through the research work of the Bureau of Standards and is now manufactured in this country under the trade names "Usalite" and "Impervite." These two porcelains have a melting point above that of platinum. However, they are impervious to gases only when glazed. The softening point of the glaze used is about 1300° C. If the tubes are glazed on the outside only, they are serviceable as pyrometer-protection tubes up to 1500° C. The glaze on the German tubes softens at 1200° C. The insulating tubes are not glazed. Pyrometer porcelain for use at lower temperatures is made by a number of American and foreign companies. The glaze on the Japanese porcelain softens at about 1100° C and its maximum serviceable temperature is about 1250° C. An unprotected porcelain tube suddenly thrust into a furnace at 1000° C will usually break. If it is inserted very slowly, however, there is little danger of breakage.

In permanent installation quartz and porcelain tubes and also the iron or chromel tubes of base-metal couples are frequently further protected by heavy outer tubes of fire clay, carborundum, graphite, etc. The outer tube is usually cemented in place in the furnace wall, forming a well into which the couple is inserted. In case the outer tube introduces too large a temperature lag or where there is danger of lowering the temperature of the hot junction of the couple by conduction of heat through the heavy tube, the latter is made open at both ends The couple and its smaller protecting tube are so mounted that the hot junction projects a few centimeters beyond the end of the outer protecting tube.

Carborundum (Fig. 53, No. 1).—Carborundum or silicon carbide, *SiC*, is used for outer protecting tubes. It has a very high thermal conductivity, low coefficient of expansion, and a high mechanical strength. When heated in an oxidizing atmosphere, slow oxidation occurs, which does not become disadvantageously

rapid at 1500° C. Carborundum without clay binder is infusible and, if heated sufficiently, decomposes. Within the carborundum tube the atmosphere is made reducing by the products of slow decomposition of the tube, and porcelain is acted upon so that the life of the porcelain tube is materially reduced. This action may be minimized by permitting ready access of air into the open end of the carborundum tube. At the higher temperature ranges carborundum is unsuitable as a protection tube, particularly for glazed porcelain not vitrified. Ordinary furnace gases do not act upon carborundum. Fused alkali or alkali carbonates, basic slags, and most metals and metallic oxides

FIG. 53.—*Protecting tubes for couples*

react with it at high temperatures. Carborundum tubes made with a refractory clay binder have slightly different characteristics, a lower thermal conductivity, higher coefficient of expansion, and are somewhat less refractory, although still possessing a softening point much above the temperature range suitable for couples.

Carborundum tubes have been used with some success in molten glass and open-hearth slag.

Nichrome or Chromel (Fig. 53, No. 2).—Cast nichrome or chromel tubes are used very extensively for protection of both base-metal and rare-metal couples. These tubes resist oxidation remarkably well, and, although much more costly than iron tubes, in many processes their longer life warrants and requires their use. Chromel "A" may be used continuously up to 1200° C. The tubes are heavy enough to withstand considerable mechanical

strain. It has not been possible as yet to draw these alloys into tubes. The material can be machined, but with difficulty, and threads may be cut for pipe fittings. In order to economize on the use of these materials, iron tubes are frequently employed having short tubes of nichrome or chromel welded to them. Only the part of the protecting tube exposed to the furnace need be of the expensive alloy. In processes carried out at low temperatures, where either iron tubes or chromel and nichrome tubes may be employed, experiments should be performed to determine the relative values of these tubes in life in hours per dollar of cost. There are practically no data available in this regard. In heat-treating furnaces and carbonizing furnaces chromel "A" is often used. Chromel "C" and nichrome last many months in lead baths. Chromel and nichrome do not volatilize as readily as iron. Base-metal couples are thus better protected by these tubes than when iron or steel is employed. Chromel "A" contains practically no iron. Chromel "C" and nichrome have a rather high iron content.

Graphite (Fig. 53, No. 3).—Graphite tubes afford an excellent protection to quartz or porcelain tubes on rare-metal couples and are frequently used with base-metal couples for molten metals. Porcelain incased in a sheath of graphite can be used in molten aluminum. Platinum couples must be thoroughly protected against the vapors distilled from graphite or carbon and from the reducing atmosphere present near heated graphite.

Clay (Fig. 53, No. 4).—Fire-clay outer protecting tubes are used for protection in kilns, glass and steel furnaces, annealing ovens, etc. Usually they are mounted vertically in the top of the furnace and may be cemented in place. Small fire-clay insulating tubes are used on base-metal couples.

Corundite (Fig. 53, No. 5).—Corundite consists of emery with a plastic clay binder. It is used in ceramic and glass industries for outer protecting tubes.

Alundum.—Natural corundum usually contains a large amount of iron. The artificial product, fused Al_2O_3, known under the trade name of "Alundum," is practically free from iron. This material is very desirable for protecting rare-metal couples and for use also as outer protecting tubes. The tubes are made from ground alumina mixed with a clay binder. The more refractory the tubes, the less impervious to gases they become. The inner protecting tubes are glazed in order to reduce the porosity, and

the glaze is coated with an outer layer of alundum. This method of preparation permits the use of the tubes at temperatures above the softening point of the glaze. Such tubes are serviceable up to 1400° C. Outer protecting tubes without glazing are made to withstand temperatures up to 1550° C, and even higher. Alundum is mechanically strong and resists temperature changes much better than porcelain.

Nickel.—In an oxidizing atmosphere a thick tough coating of the oxide forms on nickel which does not readily scale. The oxide thus affords protection against further corrosion.

Pure nickel is frequently used for cyanide baths. It is very important, however, for this purpose, that the nickel be free from oxides and other impurities soluble in cyanide.

Steel and Iron.—Seamless steel and wrought-iron tubes are usually furnished with base-metal couples. They are satisfactory for many processes up to 800° or 900° C; for example, in a muffle furnace.

Calorized Iron.—Calorizing is a process by which the surface of a wrought-iron tube is impregnated with metallic aluminum. Calorized tubes resist oxidation better than the pure iron or steel tubes.

Duriron.—Duriron, a high silica iron alloy, is sometimes used at lower temperatures as a protection against acid fumes. When subjected to sudden temperature changes, the material may fracture.

Chromon.—An alloy of 75 per cent iron and 25 per cent chromium, useful for molten brass and bronze. A light protecting tube may be used for 100 to 200 determinations of less than one minute each.

IV. GENERAL THEORY OF OPTICAL AND RADIATION PYROMETRY

The temperature of a material may be obtained from a measurement of the intensity of the radiant energy it emits. This measurement may refer to the radiation of all wave lengths emitted by the material, or, if the material is glowing, the measurement may refer to the visible light emitted or to the radiation in a very restricted portion of the visible spectrum. However, in general, the intensity of radiation depends not alone upon the temperature of the source, but also upon the particular material constituting the source. Thus glowing carbon appears to the eye about three times as bright as glowing platinum when both are at the same

temperature. This is technically expressed by the statement that the emissive power or emissivity of carbon is about three times that of platinum. A material having the highest theoretically possible emissivity is known as a black body. In general, it is customary to assign a numerical value of 1 to the emissivity of a black body. Hence all other materials have an emissivity less than 1. A black body is experimentally realized by uniformly heating a hollow inclosure and observing the radiation coming from a small opening in the wall. The intensity of radiation emitted from this opening depends only on the temperature of the walls. It does not depend upon the material of which the walls are constructed. If E equals the emissivity of any non-transparent material and R equals its reflection coefficient, it can be shown that $E+R=1$. If a material having an emissivity of, say, 0.40, and hence a reflection coefficient of 0.60, is placed inside a black body it becomes indistinguishable from its surroundings. The total intensity of radiation leaving the material is the same as that emitted by the black body. Thus while the material actually emits only 40 per cent of the intensity of a black body at the same temperature, under the above conditions 60 per cent of the radiation falling upon it from the walls of the inclosure is reflected, with the net result that the object appears of the same intensity as its surroundings. However, if the material is removed from the black body and placed in the open air, the reflected intensity is no longer present and the object appears but 40 per cent as bright as a black body at the same temperature. Optical and radiation pyrometers are usually calibrated to read correctly when sighted upon a black body. Fortunately many technical processes are carried out under black-body conditions. Muffle furnaces, many annealing furnaces, etc., are sufficient approximations to "black bodies" to give practically correct temperature readings with the optical or radiation pyrometer. Some materials in the open are nearly "black;" for example, the oxide formed on iron and steel ingots, rails, etc. In general, however, corrections must be applied to the pyrometer readings to obtain the correct temperature of materials in the open. These corrections are very large in the case of clean molten metals. The presence of an oxide film on the molten metal surface greatly reduces the corrections. For temperature control it is not always necessary to correct the observed readings. As far as the factor of emissivity is concerned, the actual pyrometer readings, although known to be too low, will always be too low by the same amount from time to time for

the same observed temperature, and hence will furnish as good information for temperature control and uniformity as could the true temperatures.

1. OPTICAL PYROMETER TEMPERATURE SCALE

The temperature scale for the optical pyrometer is based upon Wien's law for the distribution, in the spectrum, of the energy of a black body. This law may be stated by the following equation, where λ denotes the wave length in microns, c_2 a constant = 14 350, ϑ the absolute temperature of the black body, J_λ the intensity at the wave length λ (that is, at a particular color such as red), and c_1 a constant, the value of which is of no moment in pyrometry since, as will be seen, it disappears from the actual working equation.

$$J_\lambda = c_1 \lambda^{-5} e^{-\frac{c_2}{\lambda \vartheta}} \text{ for a back body.} \tag{1}$$

The intensity of radiation J'_λ, of wave length λ, from a nonblack body of temperature ϑ and emissivity E_λ is given by equation 2.

$$J'_\lambda = c_1 E_\lambda \lambda^{-5} e^{-\frac{c_2}{\lambda \vartheta}} = c_1 \gamma^{-5} e^{-\frac{c_2}{\lambda S_\lambda}} \text{ for a nonblack body.} \tag{2}$$

In the third term of (2) we define S_λ as the apparent temperature in degrees absolute of the nonblack body. This is the temperature measured by the optical pyrometer and is less than the true temperature ϑ for all materials except black bodies, when it becomes equivalent to ϑ. From (2) we have:

$$\frac{1}{\vartheta} - \frac{1}{S_\lambda} = \frac{\lambda \log E_\lambda}{0.4343 c_2} = \frac{\lambda \log E_\lambda}{6232} \tag{3}$$

Thus knowing λ and E_λ, it is always possible to obtain the true temperature ϑ from the observed temperature S_λ.

An optical pyrometer is simply a photometer using monochromatic light (usually red) in which the intensity of radiation from either a standard or a constant source (electric lamp, oil flame, etc.) is compared with that from the object of which the temperature is desired. Frequently the two intensities are made equal by adjusting various types of absorbing devices (absorption glasses, iris diaphragms, etc.), interposed either on the furnace side or the standard lamp side of the pyrometer, depending upon which source is normally the brighter. In this process of comparison the term $c_1 \lambda^{-5}$ of equation (1) is embodied as one of the calibration constants of the instrument.

2. RADIATION PYROMETER TEMPERATURE SCALE

The temperature scale for the radiation pyrometer is based upon the Stefan-Boltzmann law expressing the relation between the total energy J radiated per unit time per unit area by a black body and its absolute temperature, ϑ^0 abs. as follows:

$$J = \sigma(\vartheta^4 - \vartheta_0^4), \tag{4}$$

where ϑ_0 denotes the absolute temperature of the surroundings or of the measuring instrument receiving the radiation, and σ an empirical constant. In general ϑ_0^4 is negligible in comparison with ϑ^4, so the above relation becomes:

$$J = \sigma\vartheta^4. \tag{5}$$

For a nonblack body we have:

$$J' = \sigma E\vartheta^4 = \sigma S^4, \tag{6}$$

where E is the total emissivity and S is the apparent absolute temperature of the object sighted upon as measured by the radiation pyrometer. From (6) one obtains:

$$E = \frac{S^4}{\vartheta^4} \text{ or } \log E = 4\ (\log S - \log \vartheta). \tag{7}$$

Thus, knowing the total emissivity E of any material, it is possible to obtain the true temperature ϑ from the apparent temperature S as measured by a radiation pyrometer.

Summary statement of the two radiation laws: Equation (1) states that the intensity of radiation of a fixed wave length from a black body is proportional to $e^{\frac{-\text{constant}}{\vartheta}}$

Equation (5) states that the total radiation of all wave lengths emitted by a black body is proportional to ϑ^4. These two laws, which form the basis of optical and radiation pyrometry respectively, are in agreement with the temperature scale defined by the gas thermometer up to 1550° C, the upper limit at which a gas thermometer has been used satisfactorily. Above this range to 2500° C the scales defined by these two laws have been found, experimentally, to be in mutual agreement, and it is believed that they correctly represent the thermodynamic scale for all temperatures.

V. OPTICAL PYROMETRY

1. FERY OPTICAL PYROMETER

Fig. 54 illustrates the principle of the Fery optical pyrometer. G is a means for producing a divided photometric field. In the later instruments a Lummer-Brodhun or silver-strip cube is employed. Part of the field of view is illuminated by the source sighted upon and part by the gasoline lamp L, which burns at a constant brightness. By moving the wedges of black glass, pp', the thickness of absorbing glass in the line of sight can be varied until the part of the field illuminated by the source has the same brightness as that illuminated by the lamp. A red glass screen is used in the ocular, so that fairly monochromatic light of this color (0.65μ to 0.63μ) is compared. The relation between the thickness of the wedges x read on a scale and the absolute temperature ϑ is $x-P=-Q/\vartheta$ where P and Q are positive constants determinable by two calibration points. The instrument must be focused upon the radiating source, but no corrections for sighting distance need be applied. The Le Chatelier optical pyrometer designed earlier is similar in principle, but is not of constant aperture, and important corrections must be made with change of focus.

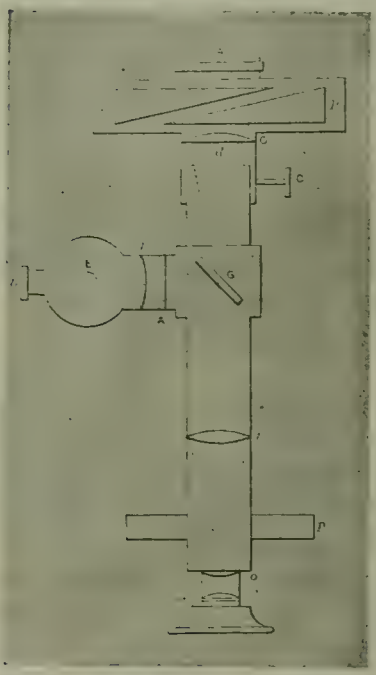


FIG. 54.—*Types of optical pyrometers*

In this pyrometer photometric match is obtained by the use of absorbing wedges

Brief directions for use of Fery optical pyrometer: Fill standard lamp about two-thirds full of gasoline (or amylacetate if originally calibrated for this material) and adjust flame until tip burns on a level with the top of the slit cut in the tube incasing the lamp. Focus eyepiece by drawing in or out until the field illuminated by the lamp is well defined. Focus the objective by thumbscrew C until the source sighted upon is clearly outlined. Turn into the field the red glass screen in the eyecup and match the two photometric fields by adjusting the thumbscrew which moves

the wedges. Observe the reading on the scale. This reading is converted into temperature by use of a table or plot of scale reading versus temperature furnished with the instrument. For varying and extending the range of the instrument two removable absorption glasses are used—one, A', on the standard lamp side and one, A, immediately in front of the adjustable wedges. In making observations care must be taken to note whether these glasses are in or out, and in converting scale readings to temperature, to observe that the correct table or plot corresponding to the particular combination employed is used.

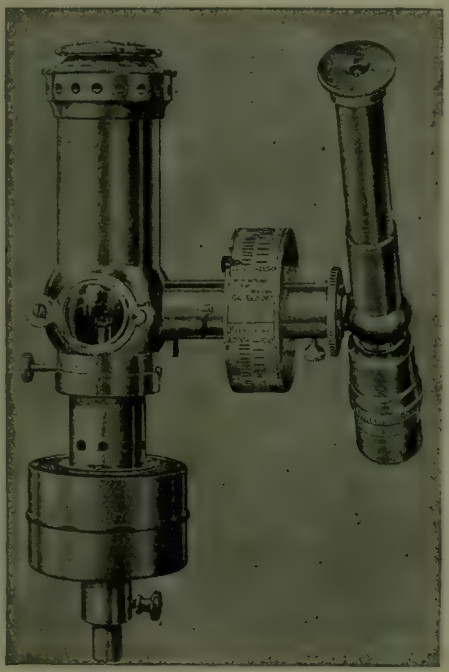


FIG. 55.—*Types of optical pyrometers*

This instrument employs a principle similar to that shown in Fig. 54

2. SHORE PYROSCOPE

The Shore pyroscope, Fig. 55, operates upon a principle very similar to that of the Fery optical pyrometer. The instrument has a scale graduated to read temperature directly, which is a material advantage. The design of optical parts is rather unnecessarily complicated, and it is difficult to match the two fields on account of color differences. The pyroscope is used extensively and with satisfactory results where high precision is not required.

(*a*) DIRECTIONS FOR USE OF SHORE PYROSCOPE.—Fill lamp two-thirds full of kerosene oil. Adjust the flame to burn at a height of about three-fourths of an inch. Focus on source by turning knurled ring on end of telescope tube. Turn knob by side of scale until the inner and outer fields match in brightness. Read temperature of source directly from scale setting.

3. WANNER PYROMETER

Fig. 56 illustrates the arrangement of the optical parts in the Wanner pyrometer. The comparison light is a 6-volt incandescent lamp illuminating a glass matt surface in front of the slit S_2. The slit S_1 is illuminated by the source sighted upon. Light from each slit passes through the collimating lens O_1, the direct vision spectroscope P, a Wollaston prism R, a biprism B, the second collimating lens O_2, and is brought to a focus at F. The Wollaston prism produces two images of each slit which are polarized at right angles to each other. The biprism again doubles the number of images, so that there are finally four images of each slit at F. Six of these images are diaphragmed off by the screen D. The two remaining images, one of each slit, are superposed and are polarized at right angles to each other. From this point the light passes through the nicol prism A and the ocular lens E. The direct-vision spectroscope is so adjusted that only red light of wave length about $\lambda = 0.65\mu$ reaches the eye, the other colors

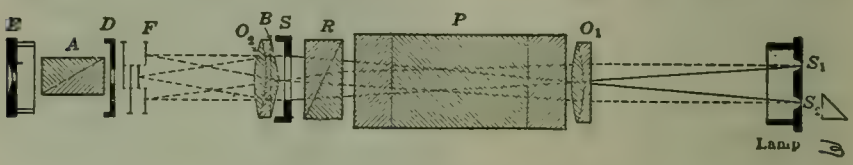


FIG. 56.—*Types of optical pyrometers*

In this instrument photometric match is obtained by the use of a polarizing system

being diaphragmed off by the screen D. The ocular is focused on the dividing edge of the biprism B. The eye perceives a circular photometric field, half of which is illuminated by the slit S_1 and half by the slit S_2. The light from the two fields is plane polarized and the plane of polarization in one field is at right angles to the plane of polarization in the other field. Consequently, on rotating the nicol prism A, one field increases and the other field decreases in intensity. A setting is obtained when the two fields match.

In order to determine the proper brightness at which to operate the electric lamp illuminating the slit S_2, the pyrometer is sighted on a source of standard brightness. This consists of an amyl-acetate lamp with a flame gage having a window of ground glass. The flame is adjusted to a specified height. The ground glass window illuminates the slit S_1. The analyzer nicol A is set at specified normal point or angle marked on the instrument. The current through the electric lamp is then varied by means of a

rheostat until the two fields are matched, and the current is read from the ammeter. This process should be repeated several times and a mean value of the current settings obtained. In using this instrument the current is adjusted to this mean value. The electric lamp burns at a high temperature and consequently deteriorates noticeably. Hence the above adjustment on the normal point requires frequent redetermination. For high precision the adjustment should be made both before and after a series of temperature readings. In the industrial plant once a day or once a week is sufficient, depending upon the amount of use.

The calibration of the instrument follows the law

$$\log \tan \varphi = a - b/\vartheta,$$

where φ is the angular reading of the analyzer, ϑ the absolute temperature, and a and b positive empirical constants. The relation between $\log \tan \varphi$ and $1/\vartheta$ is linear. Two calibration points serve to determine a and b and a table or plot may be made of φ versus $t°$ C. Usually such a table is furnished with the pyrometer, or the instrument may be sent to the Bureau of Standards for calibration.

The instrument described above is satisfactory for temperatures greater than 900° C. In the temperature range 700 to 900° C the intensity of light from the furnace sighted upon is insufficient to permit accurate settings. Hence for temperatures from 700° up the direct vision spectroscope P is replaced by a red glass screen, or the objective lens O_1 is made of red glass, and the slits S_1 and S_2 are of much wider opening.

On account of stray light the Wanner pyrometer is not accurate at very small or very large angular readings. Moreover, the temperature increases so fast at large angles that the angles would have to be observed with extreme precision in order to obtain any accuracy when expressed in degrees of temperature. The range of the instrument is thus confined to from about 10 to 80 angular degrees.

Table 14 illustrates the relation between angular reading and temperature for a particular instrument. Different instruments may have entirely different or practically identical calibrations, as desired, depending upon the adjustment of the normal point.

TABLE 14.—Relation Between Angular Readings and Temperature for a Particular Instrument

Angle	Temperature		Angle	Temperature	
	Without absorption glass	With absorption glass		Without absorption glass	With absorption glass
	°C	°C		°C	°C
10	900	1390	50	1185	2020
20	990	1575	60	1255	2200
30	1060	1725	70	1360	2460
40	1120	1865	80	1535	3030

Thus the above instrument is satisfactory for temperatures up to 1500° C. For higher temperatures an absorption glass is mounted in front of the slit S_1, which decreases the light from the furnace in a known ratio. The third column shows the temperatures which may be measured when an absorption glass of suitable density is employed. If still higher temperatures are desired a denser absorption glass must be used.

With the Wanner pyrometer the tip of the flame of the amyl acetate lamp should burn level with the top of the flame gage. The setting on the normal point is tedious work, as the flame flickers over the field. A screen of black paper placed around the lamp helps to reduce the flicker. The observations must be made in a closed room free from drafts. Any error in the setting of the normal current is carried over to the final temperature measurements, so that it is exceedingly important to exercise all possible care in these preliminary adjustments. Examine the screen of the flame gage to assure that no smoke has deposited upon it. A slight film of smoke from the lamp may cause an error of 100° or more. The amyl acetate used in the lamp need not be of high purity. Any good grade of amyl acetate is satisfactory.

From experience with several hundred instruments in use in the technical industries it is evident that these pyrometers are subjected to great abuse. The instrument is made up of delicate optical parts and should not be allowed to become heated. Many of the parts are set in wax and the various optical surfaces are cemented by Canada balsam. The Wollaston prism and the nicol prism in the rotating eyepiece are made of calcite. This material is very soft, and, although it has the appearance of glass, may be scratched with the finger nail. In practically half the instruments examined these parts have been deeply cut by knives

or pointed steel tools. All persons using this pyrometer should be cautioned not to touch any optical part except the lens in the eyecup, which requires occasional cleaning. Do not change the setting of any screw. The moving of a single screw may throw out the adjustment of the pyrometer and cause errors of 500°. If the position of any screw on the body of the instrument is altered, do not attempt to readjust the instrument, but return it to the maker. The replacement of the electric lamp will not alter the calibration of the pyrometer.

4. SCIMATCO PYROMETER

Fig. 57 illustrates the Scimatco pyrometer, an improved form of the Wanner pyrometer. All but one of the screws, the tampering with which affects the calibration of the instrument, are inclosed in a metal sheath, and hence are not readily accessible. The instrument has both an angular scale and a scale graduated directly in degrees of temperature. The box at the left contains a 6-volt storage battery, an ammeter, and an adjustable rheostat. For obtaining the proper setting of the current the pyrometer is clamped in its carrying case. The amyl acetate lamp and flame gage are so mounted that the ground glass of the gage is directly in contact with the glass window of the pyrometer, opening to the slit S_1 of Fig. 56. The tip of the flame is adjusted until it is just visible on looking through the bottom of the red glass window in the dial of the instrument. With the Wanner or Scimatco pyrometer the observer can not see through the instrument the object sighted upon. This may cause inconvenience if it is desired to measure the temperature of a small crucible in a furnace.

FIG. 57.—*Types of optical pyrometers*

This instrument employs a principle similar to that shown in Fig. 56

5. F AND F PYROMETER

Fig. 58 illustrates the arrangement of optical parts in the F and F pyrometer. Light from the furnace is focused at the center of the silver strip cube *C*. This cube produces a circular field divided through the middle. One half of the field receives light from the furnace and the other half from the ground-glass

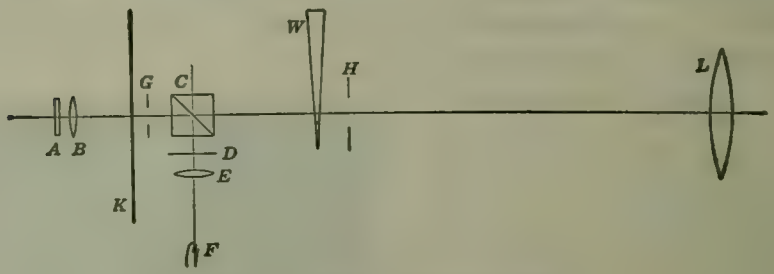


FIG. 58.—*Types of optical pyrometers*

In this instrument photometric match is obtained by the use of an absorbing wedge

screen *D*, which is illuminated by the electric lamp *F* through the condenser lens *E*. The ocular containing the red glass screen *A* and lens *B* is focused on the dividing edge of this photometric field. *G* and *H* are diaphragms which limit the cone of rays employed. The two fields are matched by turning a thumbscrew,

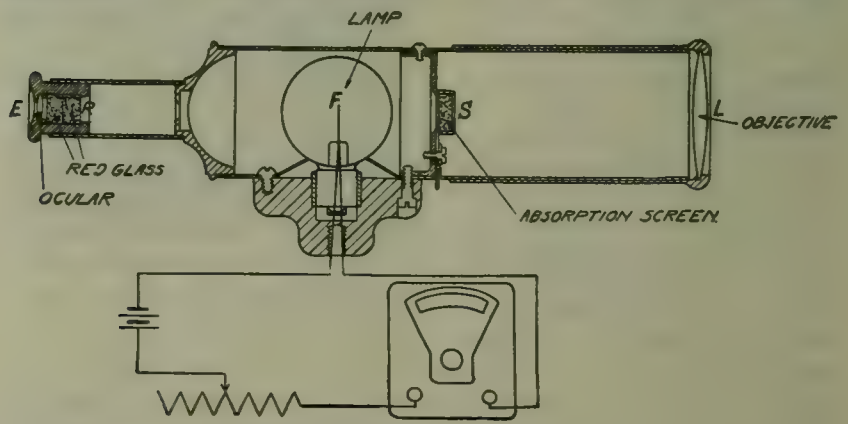


FIG. 59.—*Types of optical pyrometers*

Optical system and electric circuits of disappearing filament type of optical pyrometer.

which moves the black glass wedge *W* across the path of the light from the furnace. By a system of gears this movement is transferred to a circular scale on the dial *K* of the instrument. In appearance the pyrometer resembles the Scimatco, and is used in the same manner. For a normal-point setting the pointer is adjusted

to read the normal angle and after removing the tube carrying the lens L the instrument is clamped in its case. The flame gage of the amyl acetate lamp is so mounted that its ground-glass window is adjacent to the diaphragm H. A table is furnished with the instrument giving the relation between the scale reading in angular degrees and degrees of temperature, or a temperature scale is engraved upon the dial. This instrument is so designed that the object sighted upon is clearly imaged, a distinct advantage over the Wanner pyrometer. The relation between the scale reading α and the absolute temperature ϑ is $\alpha - P = -Q/\vartheta$ where P and Q are positive constants determinable by two calibration points.

A more recent improvement of this pyrometer consists in the use of a semicircular instead of linear wedge. A semicircular disk is so ground that the thickness decreases linearly along the straight

FIG. 60.—*Types of optical pyrometers*

Disappearing filament type

edge. A compensating wedge of clear glass is cemented to the absorbing glass to correct for refraction. The comparison lamp is mounted in the line of sight, replacing the cube C, Fig. 58, and the brightness of the filament, burning at a constant specified current, is matched against the source sighted upon by rotating the semicircular wedge. The relation between angular scale reading and temperature has the form: $\text{Cos } \alpha - P = Q/\vartheta$.

6. DISAPPEARING FILAMENT OPTICAL PYROMETER

The disappearing filament optical pyrometer was first devised by E. F. Morse (U. S. Letters Patent Nos. 696878; 696916). Figs. 59 and 60 illustrate the Leeds & Northrup form of this instrument.

The filament of a small electric lamp F, Fig. 59, is placed at the focal point of an objective L and ocular forming an ordinary telescope, which superposes upon the lamp the image of the source viewed. Red glass, such as Corning "high-transmission red," is mounted at the ocular to produce approximately monochromatic light. In making a setting the current through the lamp is adjusted by means of a rheostat until the tip or some definite part of the filament is of the same brightness as the source viewed. The outline or detail of this section of the filament is then indistinguishable from the surrounding field. This is illustrated by Fig. 61. In the third setting the central portion of the filament vanishes against the background. The current is read on an ammeter and the corresponding temperature is obtained from a plot or table of current versus temperature. The empirical relation between the current i through the lamp and the temperature t°C is of the form:

$$i = a + bt + ct^2$$

where a, b, c, are constants requiring for their determination at least three standardization points.[14]

The lamps should not be operated at temperatures higher than 1500° C on account of deterioration of the tungsten filament. If this temperature is not exceeded, the calibration of the lamp is good for hundreds of hours of ordinary use. For higher temperatures absorption glasses, S, Fig. 59, are used between the lamp and the objective, or in front of the objective, to diminish the observed intensity of the source. The relation between the temperature of the source ϑ° abs. and the observed temperature ϑ_0° abs., measured with the absorption glass interposed, is as follows:

$$\frac{1}{\vartheta} - \frac{1}{\vartheta_o} = A$$

where A is for most practical purposes a constant.

Usually the instrument is furnished with a table showing the relation between the current through the lamp and the temperature both with and without the absorption glass. If, however, this relation is not given when the absorption glass is used, it may be readily determined by measuring the constant A in the above formula. To do this, sight without the absorption glass on a muffle furnace or any uniformly heated furnace at 1200° to 1500° C

[14] See section on standardization.

and observe the temperature ϑ in degrees absolute. Then, with the absorption glass in place, match the filament again and observe to what temperature ϑ_o in degrees absolute the current through the lamp corresponds. The difference in the reciprocals of these two temperatures is the constant A, which is usually of the order of magnitude -0.00015. This determination should be repeated several times and at several different temperatures of the furnace. The separate values of A should not differ by more than 1 per cent, and the mean value is used for computing the relation between the observed absolute temperature with the absorption glass and the true temperature of the source. In making these computations care must be exercised that all temperatures are converted to degrees absolute. Table 15 illustrates the calibration of a certain pyrometer, both with and without the absorption glass. The constant of this particular absorption glass has the value $A = -0.000280$. By use of this glass temperatures as high as 2730° C can be measured, although the temperature of the lamp does not exceed 1360° C.

FIG. 61.—*Appearance of field of view when adjusting the current through the lamp of the disappearing filament pyrometer*

It will be noted that the range of currents required is small, from, in general, about 0.3 to 0.6 ampere. Thus, if the ammeter is designed to give full scale deflection on 0.6 ampere, nearly half of the scale, from 0 to 0.26 ampere, is never used. The depressed zero ammeter now available meets this objection. The

moving-coil system, including the supports, pivots, and pointer, may be adjusted relative to the magnet by turning a lever on the case of the instrument to one of two positions. In one position the pointer is adjusted *on open circuit*, so that it falls over the first graduation on the scale. This adjustment is similar to the ordinary zero adjustment on any ammeter. In the second and working position the zero is depressed off the scale an amount equivalent to 0.26 ampere. The lowest scale reading is, accordingly, 0.26, and the entire scale from 0.26 to 0.60 ampere is thus utilized for the range of the pyrometer lamp.

TABLE 15.—Typical Calibration of Lamp

Current, in amperes	Temperature		Current, in amperes	Temperature	
	Without absorption glass	With absorption glass		Without absorption glass	With absorption glass
	°C	°C		°C	°C
0.26	634	943	0.36	[illegible]	[illegible]
.28	765	1190	.38	1113	[illegible]
.30	[illegible]	1386	.42	1201	2237
.32	[illegible]	1555	.46	1281	[illegible]
.34	[illegible]	1710	.50	1359	2733

Fig. 62 shows the depressed zero ammeter. The batteries and the rheostat for controlling the current through the lamp are in the same case with the ammeter. This may be carried by the observer by means of the shoulder strap illustrated.

7. GENERAL USE OF OPTICAL PYROMETERS

Optical pyrometers and radiation pyrometers, described later, afford the only means yet developed for measuring temperature above 1500° C. The high-temperature scale above 1500° C is based on the extrapolation of Wien's radiation law by means of a pyrometer of the disappearing-filament type. When the instrument is especially designed for precision work, it is possible to measure a temperature difference of 0.2° C at 1500° C. The commercial form of the instrument when properly calibrated can be relied upon to 5° C. With a well-designed optical pyrometer there is a perfect color match of the two fields at all times. Hence, contrary to the general impression, color is not matched at all, but simply brightness of uniform color. A color-blind observer will obtain the same settings as a normal observer. Forsythe [15]

[15] Gen. Elec. Rev., p. 753; Sept. 1904.

has compiled data obtained with an optical pyrometer of the disappearing-filament type by six observers, none of whom had ever used an optical pyrometer before. The average variation from the mean for the six inexperienced observers was 3° C and the maximum variation, 5° C.

Although the optical pyrometer is essential for the measurement of temperatures above 1500° C, its usefulness is by no means confined to the high-temperature range. The thermocouple can not be adapted to many processes at low temperatures; for example,

FIG. 62.—*Depressed zero ammeter used with optical pyrometer shown in Fig. 60*

the measurement of the temperature of steel rails as they pass through the rolls, ingots and forgings in the open, and small sources such as a heated wire or lamp filament. The temperatures used in the above processes may be accurately measured by the optical pyrometer. The temperature of a microscopic sample of any material can be measured by a modified form of the disappearing-filament pyrometer [16]. Also in many processes a thermocouple is not so convenient to use as an optical pyrometer, especially when the temperature is not required often enough to warrant a permanent installation of thermocouples.

[16] Burgess, B. S., Sci. Papers, No. 198. (See Fig. 141 of this paper.)

One serious objection to the optical pyrometer from the industrial point of view is the fact that it has not been made automatically recording. Since a photometric match is required for every setting, the instrument necessitates the attention of an observer, although possibly a satisfactory automatic device will be developed eventually. Another objection is the introduction of the human element into the readings, thus affording an opportunity for dishonest or prejudiced settings. The observer, if he is the operator of the furnace, should be taught that the instrument is for his own assistance and that it is not to be considered as a policial measure; otherwise the measurements should be made by a disinterested party. In a plant operating several furnaces an intelligent boy can be profitably employed whose sole work is to make the rounds of the various furnaces and measure and record the temperatures.

8. BLACK-BODY AND NONBLACK-BODY CONDITIONS

Optical pyrometers are usually calibrated to read correctly when sighted on a black body. Many furnaces approximate black-body conditions very satisfactorily. In a perfect black body the details of the inside of the furnace vanish, and a piece of steel, for example, which is being heated can not be distinguished from the background. If the objects in the furnace can be distinguished but only on close observation, and if much of the detail is lost, *after they have been in the furnace some time*, it is not likely that the temperature measurement will be seriously in error. If in error at all, the observed temperature will be too high when the furnace walls are brighter than the material being heat treated and too low when the walls are less bright. This latter condition is possible if the heat supply is variable or if it is shut off and the furnace allowed to cool.

That a steel ingot placed in a heated furnace may appear much hotter than it really is is a fact not always appreciated. The surface of the ingot appears hot because it reflects the bright light from the walls of the furnace. Of course in comparison to the much greater brightness of the walls, the cold ingot appears black, but this is due to the intense contrast. If the ingot is viewed alone when the *direct* radiation from the furnace is screened from the eye, it also is bright. Thus when an optical pyrometer is sighted on an ingot in the furnace, part of the light reaching the instrument comes from the side walls and is reflected by the surface of the ingot. Iron oxide reflects about the least amount of

any material met with in metallurgical practice. Its emissivity is approximately 0.95; hence its reflection coefficient is 0.05. Suppose an iron ingot at room temperature were suddenly placed in a furnace at 1200° C. Although the surface of the ingot is cold, it reflects 5 per cent of the light falling upon it from the hot side walls. This 5 per cent of reflected radiation gives the ingot the appearance of an object at 950° C. The measurement by the optical pyrometer accordingly would be 950° C, although actually the ingot is at room temperature. The higher the reflecting power of the material the greater the observed temperature under the above conditions. Thus cold platinum would appear to be at about 1160° or at almost the same temperature as that of the furnace. One method for reducing the error due to reflected radiation is to view the object through a large open door on a surface parallel to the opening. If the door is large enough, the surface of the ingot thus loses a great portion of the furnace wall contributing the reflected light. The ingot reflects the image of the opening left by the door, and this opening is not radiating. It must be pointed out that the reflection from any material being heat treated is diffuse, like that from a matt surface, so that an opening in a furnace wall is not truly imaged, as would be the case with a polished-mirror surface. Stray reflection is, however, greatly minimized even with matt surfaces by sighting through a large door on a surface parallel to the opening. When the material has attained the temperature of the side walls, it is, of course, not desirable to open a large door, since then the opening affects the black-body conditions. The pyrometer should be sighted through a small peephole as soon as approximate temperature uniformity is obtained. A more satisfactory method for reducing the stray reflections than by opening a large door is to sight into a deep, wedge-shaped cavity or hole made in the metal being heat treated. If this cavity is deep enough, very little radiation from the side walls can be reflected from it. If such a hole can not be made conveniently, a length of iron pipe closed at one end or a porcelain tube may be placed in good thermal contact with the material and so aligned that the pyrometer may be sighted through a peephole directly into the bottom of the tube.

The effect of reflected light is very noticeable in an empty coke oven. The reflection coefficient of the brick walls is comparatively high. If a perfect mirror were placed parallel to a glowing surface, the mirror would appear as hot as the surface. This

effect of reflection takes place in a coke oven so that both walls appear of approximately equal brightness, even though they may differ considerably in temperature. Frequently a patch of the wall on one side becomes coated with a layer of coke. Since the coke has a higher emissive power than brick, this patch appears much hotter. Actually it is at about the same temperature as the less-bright surrounding wall. On account of reflection a corresponding bright patch appears on the opposite wall, although this wall may be free from coke. It is evident that the measurement of temperature of a portion of a nonuniformly heated furnace by means of an optical pyrometer is difficult unless the precautions suggested above are taken. As soon as the furnace attains temperature uniformity and equilibrium the optical pyrometer gives the true temperature very easily and readily.

When an optical pyrometer is sighted on a glowing material in the open, it reads too low. Certain materials, important industrially, have a very high emissivity, so that the corrections necessary to add to the observed temperatures to convert them into true temperatures are small. Thus with iron oxide the correction is only 10° at 1200° C. The corrections are very large for clear, molten metals, but are smaller for the oxides which soon form on the molten surface when exposed to the air. Table 16 shows the true temperatures corresponding to the temperatures observed when sighting on certain materials in the open. For temperature control it is unnecessary to apply these corrections. The observed temperatures, although known to be low, will be low by the same amount from time to time, and hence will serve just as satisfactorily for reproducing temperature conditions in any process as the corrected temperatures. The above statement must be modified if factors other than emissivity of the material require consideration. For example, reproducible results can not be expected if heavy clouds of smoke are in the line of sight one day and not on the next day. If the pyrometer is sighted on a stream of molten iron during pouring or tapping, the surface of the metal is usually clear and free from oxide. If the stream should at any time contain much slag, the surface will show bright patches on account of the higher emissivity of the slag. To make the readings conform with those taken on the clear stream, one must sight on the darker spaces between the slag or sight upon the slag and correct both sets of data according to Table 16.

TABLE 16.—True Temperature Versus Apparent Temperature Measured by Optical Pyrometers Using Red Light($\lambda=0.65\mu$)When Sighted Upon the Following Materials in the Open

Observed temperature, degrees centigrade	True temperature, degrees centigrade						
	Molten copper	Molten iron [a]	Solid iron oxide	Solid nickel oxide	Nichrome or chromel	Molten slag [b]	Bright platinum
700			700	701	702		730
800			801	802	804		861
900			902	904	906		972
950	1000		953	955	958		1030
1000	1150		1004	1007	1010		1087
1050	1213		1055	1058	1063		1144
1100	1277	1183	1106	1110	1116		1202
1150	1341	1239	1158	1162	1170		1260
1200	1405	1296	1210	1215	1224		1330
1250	1470	1353		1267			1375
1300	1536	1410		1320			1433
1400		1525				1455	1555
1500		1641				1565	1675
1600		1758				1670	
1700		1876				1780	
1750		1935				1830	

[a] Computed for $E_\lambda=0.40$, this being the best value for ordinary steel practice.

[b] Computed for $E_\lambda=0.65$, an average value for liquid slags.

The above table was computed from the following equation where ϑ is the true absolute temperature, S the observed absolute temperature, and E_λ the emissivity for the wave length λ. This wave length has been selected as $\lambda=0.65\mu$, the approximate value for optical pyrometers.

$$\frac{1}{\vartheta}-\frac{1}{S}=\frac{\lambda \log E_\lambda}{6232}=\frac{\log E_\lambda}{9588}$$

Table 17 gives the emissivity of various materials for this wave length. The change of emissivity with temperature is usually small for metals.

TABLE 17.—Monochromatic Emissivity for Red Light (λ=about 0.65μ)

Material	E (0.65 μ)	Material	E (0.65 μ)
Silver	0.07	Cuprous oxide	0.70
Gold, solid	.13	Iron oxide, 800° C	.98
Gold, liquid	.22	Iron oxide, 1000° C	.95
Platinum, solid	.33	Iron oxide, 1200° C	.92
Platinum, liquid	.38	Nickel oxide, 800° C	.96
Palladium, solid	.33	Nickel oxide, 1300° C	.85
Palladium, liquid	.37	Iron, solid and liquid	.37
Copper, solid	.11	Nickel, solid and liquid	.36
Copper, liquid	.15	Iridium	.30
Tantalum, 1100° C	.60	Rhodium	.30
Tantalum, 2600° C	.48	Graphite powder (estimated)	.95
Tungsten, 1000° C	.46	Carbon	.85
Tungsten, 2000° C	.43	Porcelain (? ?)	.25 to .50
Tungsten, 3000° C	.41		
Nichrome, 600° C	.95		
Nichrome, 900° C	.90		
Nichrome, 1200° C	.80		

Table 18 shows the corrections which must be added to the readings obtained with an optical pyrometer using light of wave length $\lambda = 0.65\mu$, for various emissivities, in order to obtain the true temperatures. These data are especially useful when carefully plotted on a large scale, similar to Fig. 63.

TABLE 18.—Correction to Observed Temperature for Pyrometer Using Red Light (λ=0.65μ, c_2=14350)

Emissivity	Add corrections below for the following observed temperatures, degrees centigrade										
	700	800	900	1000	1100	1200	1300	1400	1600	1800	2000
0.30	55	67	80	95	111	129	148	168	213	264	322
.40	41	50	60	71	83	96	110	125	158	195	237
.50	31	37	45	53	62	71	82	93	117	144	175
.60	22	27	33	39	45	52	59	67	85	104	126
.70	16	19	23	27	31	36	41	47	59	72	87
.80	10	12	14	17	19	22	25	29	36	44	54
.90	5	6	7	8	9	10	12	14	17	21	25
1.00	0	0	0	0	0	0	0	0	0	0	0

9. TEMPERATURE OF GLOWING GAUZE

An interesting application of the optical pyrometer is for the measurement of the temperature of gauze electrically or otherwise heated. In certain chemical processes platinum gauze electrically heated is used as a catalyzing agent, and must be maintained at a constant temperature. This is readily done by sighting normally on the surface of the gauze with an optical pyro-

meter. The observed temperatures may be thus exactly reproduced from day to day. If it is required to convert the observed temperatures into exact true temperatures of the wire forming the gauze the problem is difficult. An approximate solution satisfactory for all industrial work is, however, easily obtained.

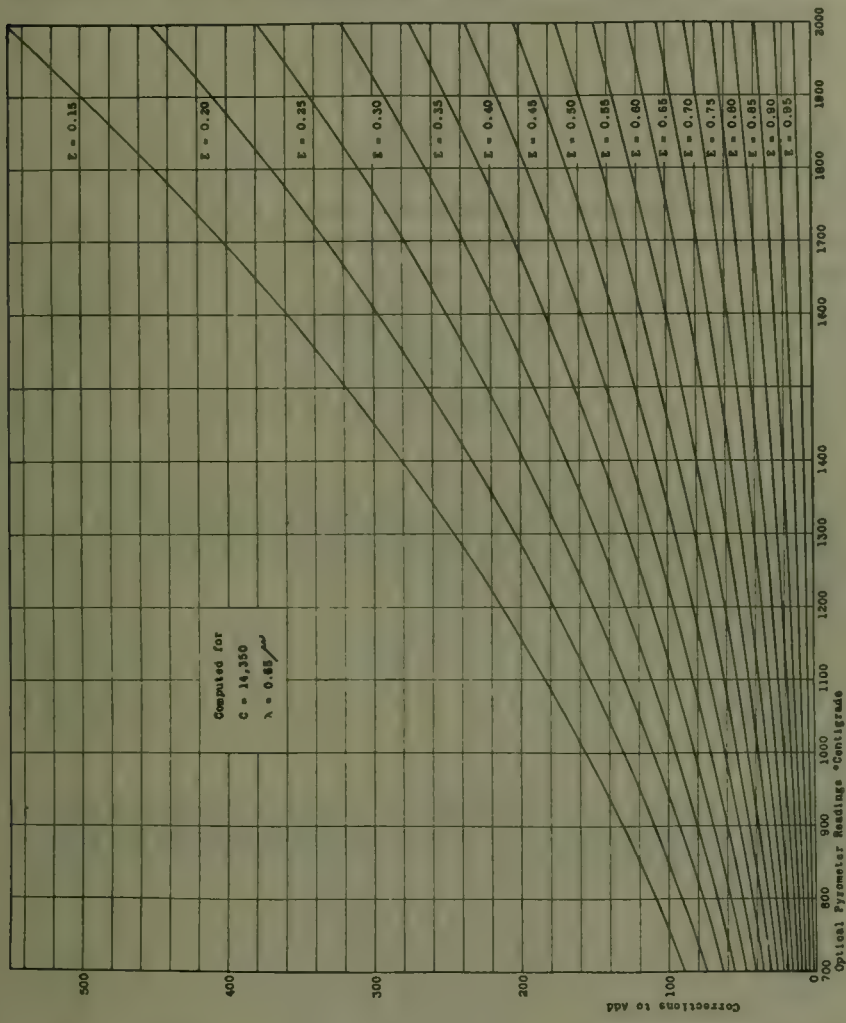


FIG. 63.—*Correction, for emissivity, to temperature observed with optical pyrometer*

We will assume that the mesh of the gauze is sufficiently coarse that multiple reflection between the separate wires is negligible. Let A_1 equal the fractional part of the total area of the gauze comprised by the wire, and A_2 equal the fractional part of the total area representing the space between the wires. Let E_λ equal the emissivity of the metal employed, and E'_λ equal the effective

emissivity of the gauze as a whole; that is, taking into consideration the spaces between the wires which, of course, are not radiating surfaces. The following equations are readily apparent:

$$E'_\lambda = \frac{A_1}{A_1 + A_2} E_\lambda = A_1 E_\lambda, \text{ since } A_1 + A_2 = 1.$$

$$\frac{1}{\vartheta} - \frac{1}{S} = \frac{\log A_1 E_\lambda}{9588}$$

where ϑ is the true absolute temperature of the wire of the gauze and S is the absolute temperature observed with an optical pyrometer sighted normal to the surface.

A platinum gauze commonly employed is No. 80 mesh (80 wires to the inch) of 0.003-inch wire. For this gauze $A_1 = 0.42$. The emissivity of bright clean platinum is 0.33. The platinum of this gauze soon becomes somewhat corroded. Possibly an emissivity of, say, 0.4 is more nearly the correct value under these conditions. Hence the effective emissivity of the gauze $= A_1 E_\lambda = (0.42)\ (0.4) = 0.17$.

Usually the gauze must be viewed through a glass window. A thin glass window (see below) transmits about 90 per cent of the light falling upon it. Hence the final effective emissivity using a glass window $= E'_\lambda = (0.17)\ (0.90) = 0.15$.

The following table was computed by the formula $\frac{1}{\vartheta} - \frac{1}{S} = \frac{\log 0.15}{9588}$. A similar table for other gauzes may be computed in the manner outlined.

TABLE 19.—Platinum Gauze No. 80 Mesh, 0.003-inch Wire. Temperatures Observed Through One Window by Optical Pyrometer Sighted Normal to Surface of Gauze, versus True Temperature of Gauze.

Observed temperature	True temperature
°C	°C
[illegible]	675
650	730
700	790
750	[illegible]
[illegible]	910
[illegible]	975
900	1035
[illegible]	1095
[illegible]	1160
[illegible]	1220

10. USE OF A WINDOW

It is frequently necessary, especially in the laboratory, to sight an optical pyrometer into a furnace through a window. What correction must be applied to the observed temperatures to take account of the loss of light at the window? Kanolt has measured the transmission coefficient for a number of ordinary glass windows at $\lambda = 0.65\mu$ and obtained a mean value of 0.904. Hence we have

$$\frac{1}{\vartheta} - \frac{1}{S} = \frac{\log 0.904}{9588} = -0.0000046,$$

where θ is the true absolute temperature of the source and S is the observed absolute temperature. Table 20 is computed from the above formula.

TABLE 20.—Correction to Observed Temperatures for Absorption of Light by a Single Clean Window

Observed temperature	Correction to add
°C	°C
600	3.5
800	5.4
1000	8
1200	10
1400	13
1600	16
1800	20
2000	24
2500	36
3000	50

11. FLAMES AND SMOKE

The optical pyrometer can not be used satisfactorily when sighted through flames or smoke. Usually the presence of dense flames increases the temperature reading and the presence of smoke clouds absorbs so much radiation that the pyrometer may read several hundred degrees low. The optical pyrometer can be used to measure the temperature of the slag in an open-hearth furnace, but the flames prove a serious hindrance except during reversals, when observations may be taken to advantage. In a cement kiln the dust, smoke, and flames all combine to make the observations very untrustworthy. Carbon dioxide, water vapor, and other invisible gases produce no effect.

12. METHOD OF SIGHTING INTO A CLOSED TUBE

In many processes where smoke can not be eliminated or where black-body conditions are not satisfactory, a porcelain or other refractory tube with a closed end is inserted into the furnace. The pyrometer is sighted into this tube, which, if fairly uniformly heated over a sufficient area, affords an excellent black body. This method has been employed also for obtaining the true temperature of molten metals, but suitable refractory tubes for many molten metals have yet to be developed.

VI. RADIATION PYROMETRY

An optical pyrometer measures the intensity of a narrow spectral band of radiation emitted by a glowing object. The radiation pyrometer measures the intensity of all wave lengths, the light rays and the heat rays combined. Usually the energy of all wave lengths radiated by the source is focused in some manner upon the hot junction of a small thermocouple. The temperature to which this junction rises is approximately proportional to the rate at which energy falls upon it, which in turn, by the Stefan-Boltzmann law, is proportional to the fourth power of the absolute temperature of the source. The rise in temperature of the hot junction of the couple generates a thermoelectric emf. Hence the calibration of a radiation pyrometer consists in determining the relation between the emf developed and the temperature of the source sighted upon. This relation follows the law $e = a\vartheta^b$ where ϑ is the absolute temperature of the source, e is the emf developed by the instrument, and a and b are empirical constants determinable by two standardization points. The emf may be measured by a potentiometer or galvanometer or by any of the methods discussed under thermoelectric pyrometry. The galvanometer should have as high a resistance as is consistent with the requirement of robustness. The same type of instrument is used with the radiation pyrometer as with the ordinary thermocouple. In fact, the entire discussion on galvanometric methods of measuring emf of thermocouples is equally applicable to radiation pyrometry. The temperature of the cold junction of the couple in the radiation pyrometer is not controlled. The hot and cold junctions are in fairly close proximity and hence are equally affected by changes in room temperature. The cold junction is always shaded from the heat radiated by the source sighted upon.

1. THWING RADIATION PYROMETER

Fig. 64 illustrates the principle of the Thwing radiation pyrometer. Radiation from the furnace enters the diaphragm *A* and falls upon the hollow conical mirror *K*. The hot junction *C* of

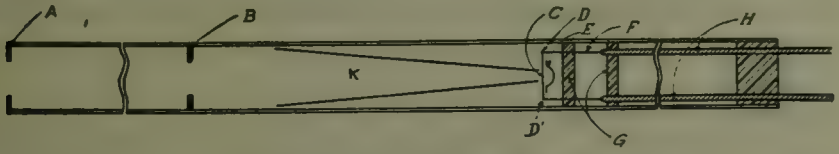


FIG. 64.—*Types of radiation pyrometers*

Receiving system of instrument employing a conical mirror

a minute thermocouple is located at the apex of the cone, and the cold junctions are at *D* and *D'*. By multiple reflection along the sides of the conical mirror the radiation is finally concentrated upon the hot junction of the couple. The emf is measured by a

FIG. 65.—*Observing the temperature of a furnace with the pyrometer illustrated in Fig. 64*

galvanometer graduated to read temperature directly. Fig. 65 shows the method of using this instrument for measuring the temperature of a large furnace. Except for incidental errors, which will be briefly considered later, the reading of the instru-

ment is independent of the sighting distance, provided the diameter of the source is sufficient to fill the cone of rays defined by the geometrical construction of the receiving tube. Fig. 66 illustrates this fact. The amount of radiation falling upon an element, D, of the conical mirror is proportional to the solid angle HDA' which is independent of the distance from the point D to the source. This is true of every point on the base of the cone

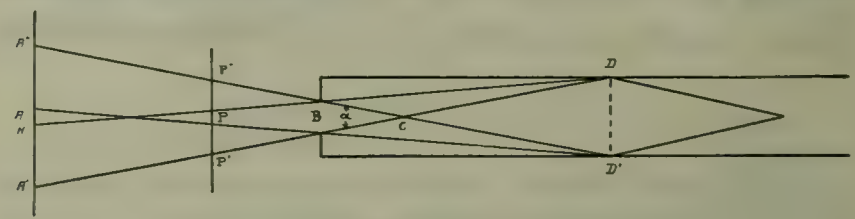


FIG. 66.—*Cone type of fixed-focus radiation pyrometer*

DD'. Hence the total quantity of energy entering the cone is independent of the distance from the pyrometer to the source, provided the source is of sufficient size. The minimum size of source for any distance is determined by the lines $A''D'$ and $A'D$. Thus for the distance BA the diameter of the source must be at least $A'A''$ and for the distance BP the diameter of the

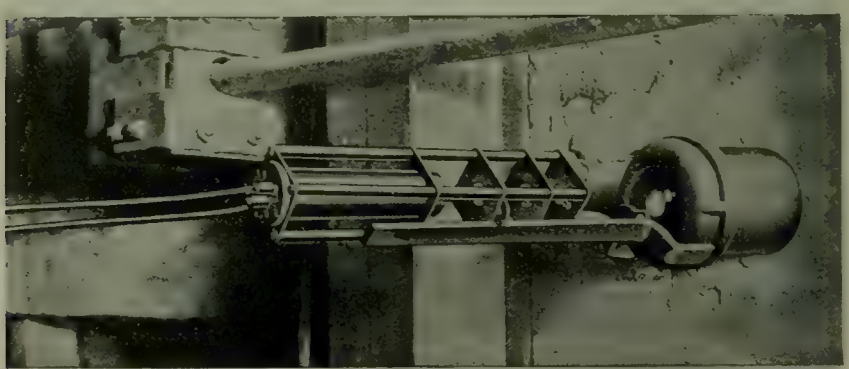


FIG. 67.—*Radiation pyrometer for fixed installation on furnace wall*

source must be at least $P'P''$. The Thwing instrument is so constructed that the source must have a diameter at least one-eighth of the distance from the source to the receiving tube. Thus at 8 feet from a furnace the opening into the furnace must be 1 foot in diameter.

For permanent installations the tube is ventilated and has several extra diaphragms to prevent local heating of the instrument and reradiation to the couple. Fig. 67 shows a perma-

nently installed Thwing radiation pyrometer sighted into the bottom of a closed metal tube which projects into the furnace.

2. FOSTER RADIATION PYROMETER

Fig. 68 illustrates the principle of the Foster radiation pyrometer. The thermocouple, *b*, and a front diaphragm, *B*, are

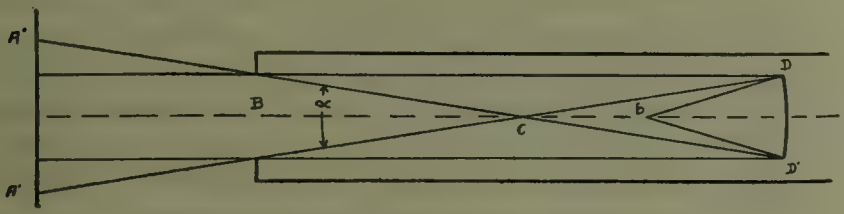


FIG. 68.—*Mirror type of fixed focus radiation pyrometer*

located at the conjugate foci of a concave mirror, *DD'*. As in the case of the Thwing pyrometer, the source must be large enough to fill the cone of rays defined by the angle α or the lines *A' CA''*. The position of the point *C* is marked by a wing nut on the tele-

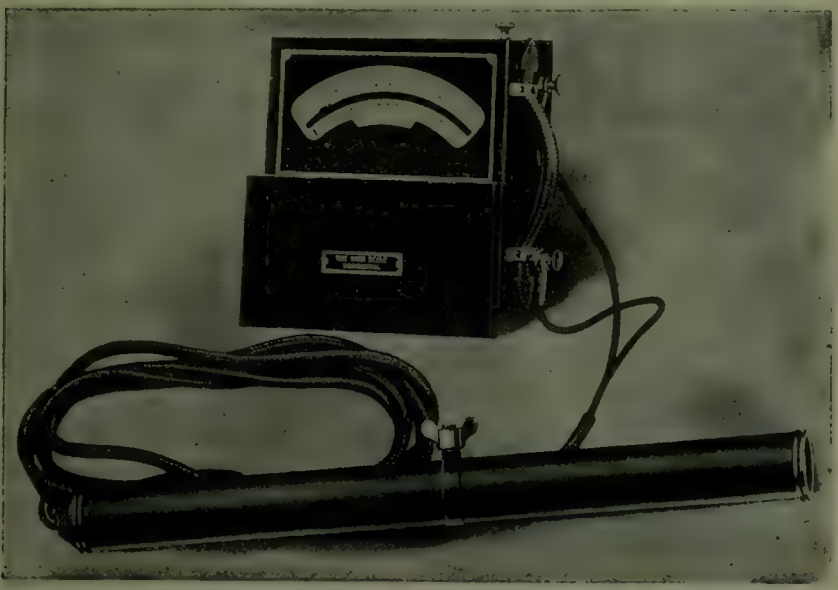


FIG. 69.—*Types of radiation pyrometers*

The receiving system of this pyrometer is shown in Fig. 68

scope tube. The angle α is made such that the diameter of the source sighted upon must be at least one-tenth the distance from the source to the wing nut. Thus for a distance of 10 feet the diameter of the opening into the furnace must be at least 1 foot. Fig. 69 illustrates the complete instrument with an indicator

graduated to read temperature directly. The Brown radiation pyrometer, which is similar in principle to the Foster pyrometer, is made with the receiving tube collapsible for convenience in carrying.

3. FERY RADIATION PYROMETER

Fig. 70 is a cross-section drawing of the Fery pyrometer made by the Taylor Instrument Cos. Radiation from the source sighted upon is concentrated by the concave mirror of speculum metal or gold, upon the hot junction of a minute thermocouple. This instrument is not fixed focus, as is the case with the other types of radiation pyrometer, but requires focusing for each sighting

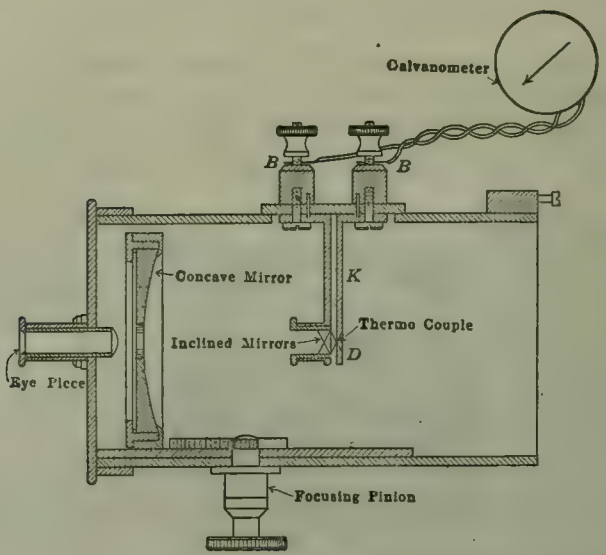


FIG. 70.—*Types of radiation pyrometer*

Mirror type of variable-focus radiation pyrometer

distance. The focusing is accomplished simply by means of an ingenious device ascribed to Fery. Two semicircular mirrors, Fig. 71 (*a*), inclined to one another at an angle of 5° to 10°, are mounted in the thermocouple box, an opening of about 1.5 mm at the center of the mirrors forming the limiting diaphragm immediately in front of the couple. The observer views by means of the telescope *V* the image of the furnace formed by the large concave mirror *MM′* and reflected by the inclined mirrors *xx′* and *yy′* through a hole in the large mirror. If the image is not correctly focused at *O*, the intersection of the two small mirrors, the image appears broken in half as shown by (*b*). Correct focus is obtained when the two halves of the image are in alignment (*c*).

Thus until correct focus is obtained certain straight lines of the source appear broken in the image. This breaking of a line is illustrated on a magnified and distorted scale by (*d*). Suppose that the pyrometer were incorrectly focused upon a line source, an arrow, the image falling at position *AB* instead of at *O*. The image of the arrow reflected from the mirror *yy'* lies at *A''B''*, and that reflected from the mirror *xx'* at *A'B'*. To the observer at *D* the projections of these images appear as two distinct arrows, thus → →. As the pyrometer is brought nearer into the correct focus by turning the pinion screw, and thus moving the large concave mirror in the direction *OD*, the points *P'* and *P''* of the reflected images move along the lines *P'O* and *P''O*, coinciding at *O* when the correct focus is obtained. The two arrows are then superposed, forming a single image.

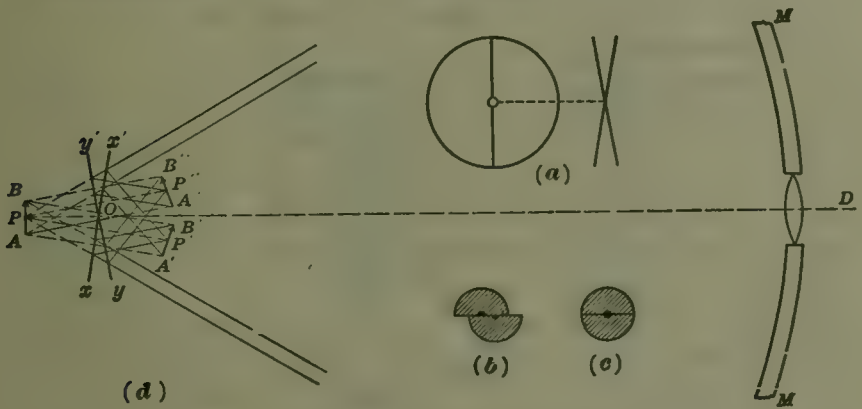


FIG. 71.—*Optical system of pyrometer shown in Fig. 70*

For the measurements of very high temperatures, usually above 1500° C the cover to the front of the telescope is provided with a sectored opening, which may be adjusted to reduce the radiation falling upon the receiver by any definite amount, and in this manner the upper temperature range of the instrument is practically unlimited. This adjustment is made by the manufacturer and should not be altered. For the lower scale range the cover is open, as shown in Fig. 72.

The readings with a Fery pyrometer, when properly focused, neglecting secondary errors discussed later, are independent of the sighting distance, as is the case with the fixed-focus radiation pyrometer.[17] The image of the source, as viewed through the small

[17] See B. S. Sci. Paper No. 250, p. 97, for the geometrical demonstration of this fact.

telescope, must cover *completely* the limiting diaphragm to the thermocouple. This diaphragm appears as a black, circular area shown at the center of the field in Fig. 71, (*b*) and (*c*). An excellent rule to follow is to sight at such a distance that the area of the image overlaps this hole and extends half way to the edge of the focusing mirrors. The Fery pyrometer requires a smaller source than the fixed focus instruments. The following table illustrates the size of source required for various sighting distances in order that the image cover the limiting diaphragm and extend about half way to the edge of the focusing mirrors, i. e. diameter of image =4 mm.

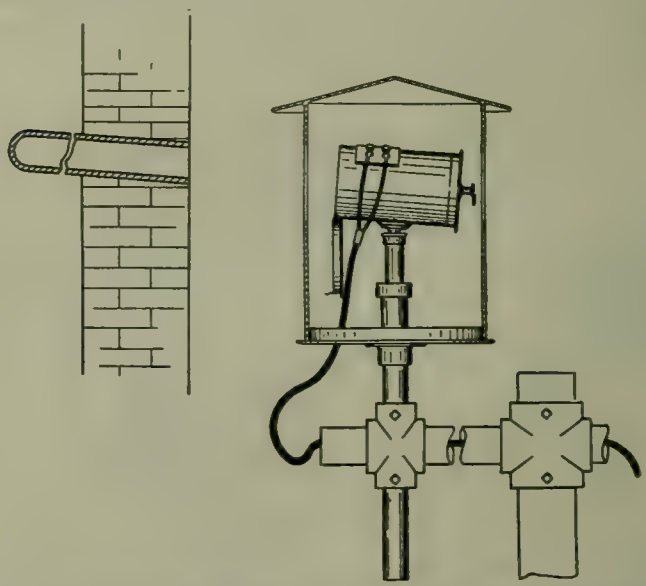


FIG. 72.—*Radiation pyrometer in weather hood, sighted into a fire-clay tube*

TABLE 21.—Size of Source versus Sighting Distance

Sighting distance	Diameter of source
Centimeters	Centimeters
70	3.2
80	3.7
100	4.8
150	8.3
200	11.2
300	16.8
500	28.5

4. ERRORS TO WHICH RADIATION PYROMETERS ARE SUBJECT

Dust and dirt allowed to accumulate upon the concave reflecting mirror may so decrease its reflection coefficient as to develop errors amounting to 100° or even 200° C. Frequently the dust can be removed from the mirror by carefully brushing with a camel's-hair brush. The mirror may be removed from the instrument and washed, but this must be done with great care to insure that the delicate thermocouple or its mounting is not disturbed. The safest practice is to take all possible precautions to prevent dust from entering the instrument. Keep the case closed or the front diaphragm of the fixed-focus instruments plugged with a cork when not in use.

As shown above, the simple theory of the radiation pyrometer states that the readings are independent of the sighting distances or size of source, provided the source is larger than the minimum size of source demanded by the geometry of the instrument. Actually this ideal condition is not always realized. Some stray radiation is reflected down the walls of the telescope case. The walls become heated by the furnace and reradiate to the couple. Limiting diaphragms are similarly heated and reradiate to the couple. For these reasons a radiation pyrometer tends to read lower the greater the sighting distance or the smaller the size of source. It is thus in general desirable to use a radiation pyrometer as nearly as possible in the same manner from day to day and to have it specially calibrated for such use. For example, if a 6-inch source be employed, decide upon a convenient sighting distance, say 4 feet, and always use this distance, and not sometimes 1 foot and sometimes 5 feet. In the Fery pyrometer both the proper size of source and the proper focusing distances are secured by following the rule suggested above, viz, focus at such a distance that the image extends halfway between the black hole and the outside edge of the focusing mirrors.

5. ADVANTAGES AND DISADVANTAGES OF RADIATION PYROMETERS

For temperatures above 1400 or 1500° C either a radiation pyrometer or an optical pyrometer must be employed. The optical pyrometer is capable of higher accuracy and is less susceptible to errors than the radiation pyrometer. Smoke and dust affect the readings of both instruments, but the radiation pyrometer is seriously affected by the presence of cooler strata of carbon dioxide and other gaseous combustion products in the furnace. Carbon

dioxide and water vapor absorb the heat rays, and hence the radiation pyrometer will read too low when sighted through such gases. The main advantage of the radiation pyrometer is the fact that it can be made automatically recording. The recording mechanism is the same as that employed for ordinary thermocouples, discussed in the section on recording pyrometry. The radiation pyrometer is desirable for many processes of lower temperature where thermocouples can not be conveniently installed. It is also useful in measuring the surface temperature of large ingots.

6. BLACK-BODY AND NONBLACK-BODY CONDITIONS

Radiation pyrometers are calibrated to read correctly when sighted upon a black body. Most furnaces approximate black-body conditions sufficiently well. When sighted on materials in the open certain corrections must be applied to the observed temperature to convert them into true temperatures. For temperature control or reproducibility the apparent temperatures

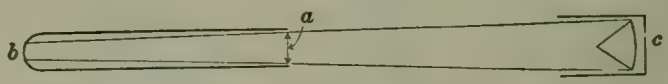


FIG. 73.—*Method for determining proper diameter of sighting tube*

may be used uncorrected, if desired, since, although known to be low, they will be low by the same amount from day to day. In case the temperature of one section of an unequally heated furnace is required, or if the furnace contains much smoke or dust, it is frequently desirable to sight the radiation pyrometer into the bottom of a fire-clay or porcelain tube, as illustrated by Fig. 72. The tube should be uniformly heated at the end for a length at least three times its diameter. The tube must have a diameter such that the cone of rays entering the telescope is not intercepted by the front of the tube. Fig. 73 illustrates this condition. Suppose the distance from the bottom of the tube to the mirror of the Fery pyrometer is 100 cm. Referring to the above table, the diameter of the source required is 4.8 cm. Lay off the distance $b = 4.8$ cm and draw straight lines from the bottom of b to the bottom of the mirror c, and from the top of b to the top of the mirror. The tube must have such a diameter at its front end a that it does not cut in on the cone of rays represented by these two straight lines. This can be determined only by actually making the above drawing to scale or by computing. It can not

be decided by looking through the telescope of the pyrometer, since the front end of the tube would not necessarily show even if it did cut in on the cone of rays. In the case of the fixed-focus instruments the diameter of the tube must be such that the cone of rays represented by the diameter $A'A''$, Fig. 66, cuts the tube in the region which is uniformly heated.

Table 22 shows the true temperatures corresponding to the apparent or measured temperatures observed with a radiation pyrometer when sighted upon various materials in the open. This table must not be confused with Table 16 for the optical pyrometer.

It will be noticed that the corrections are entirely different for the two types of pyrometer.

TABLE 22.—True Temperature versus Apparent Temperature Measured by Radiation Pyrometers when Sighted upon the Following Materials in the Open

Observed temperature, degrees centigrade	True temperature, degrees centigrade				
	Molten iron	Molten copper	Copper oxide	Iron oxide	Nickel oxide
600	.....	1130	720	630	710
650	.....	1210	775	.....	755
700	.....	1290	830	735	800
750	.....	.....	890	.....	845
800	1300	.....	945	840	895
850	1270	.....	1000	.....	940
900	1340	.....	1060	945	985
950	1410	.....	1115	.....	1030
1000	1475	.....	1170	1050	1075
1050	1550	.....	.....	.....	1120
1100	1610	.....	.....	1155	1165
1150	1680	.....	.....	.....	1210
1200	1750	.....	.....	1260	1255

VII. RESISTANCE THERMOMETRY

The operation of a resistance thermometer depends upon the variation with temperature of the resistance (mass resistivity) of an electrical conductor. A high temperature coefficient of resistivity might accordingly appear of primary importance. Certain oxides possess this property, having a resistance at room temperature from a hundred thousand to a million times that at 1000° C. However, the resistance of a suitably mounted material may be easily and accurately determined so that the consideration of high temperature coefficient is rarely, if ever, the deciding factor in the choice of the material. The fundamentally important property

is reproducibility and constancy of resistance. A further desirable feature is a simple and convenient relation between temperature and resistance. Platinum of the highest obtainable purity meets these requirements up to about 1100° C. On the basis of the reproducible temperature scale defined by the platinum resistance thermometer, when calibrated according to certain specifications, temperatures may be measured to possibly 0.1° C. at 1000° C. To obtain such precision, however, requires accurately constructed apparatus and very careful experimental manipulation.

For industrial purposes, resistance thermometers are made usually either of platinum or of nickel, the latter material being fairly satisfactory for temperatures below 300° C. In a common form of platinum thermometer about 40 cm of fine wire is wound upon a mica frame into a spiral coil about 4 cm in length and 0.7 cm in diameter. The length of the wire is so adjusted that the resistance of the coil is about 8.3 ohms at 0° C. The frame is made of crossed strips of thin mica notched at the edges to hold the wire in place. From the ends of this coil lead wires of platinum or gold are carried to the terminal head of the thermometer. The lead wires are insulated and held apart by mica disks through which the wires are threaded. The thermometer is protected by a porcelain tube glazed on the outside, or by a quartz tube which fits into the terminal head. For industrial use the refractory tube is itself protected by an outer metal tube. The metal tube is usually fitted to an extra terminal head, through which the lead wires to the line are carried. On account of the deterioration of the platinum, thermometers constructed of fine wire are not very satisfactory for industrial use above 900° C. Heavy platinum wire, 0.6 mm in diameter, may be employed up to 1100° C, but the resistance of the thermometer is so low that it is necessary to use methods of measurement which do not depend upon constancy in the resistance of the lead wires—e. g., Thomson bridge or potentiometer.

In another form of resistance thermometer a coil of fine platinum wire having a resistance of 50 ohms at 0° C is wound upon a quartz rod 3 mm in diameter and 6 cm in length and fused inside a quartz tube, so that the platinum is entirely embedded in quartz. Gold leads are carried from the coil to the terminal head. The quartz insures high electrical insulation, while still preserving excellent heat conduction. The thermometer is very compact,

and the platinum is thoroughly protected against deterioration. For work of the highest precision, in which measurements to 0.1° or 0.01° C are required, this type of thermometer is inferior to that in which the platinum is loosely mounted on a mica frame, since its calibration is slightly altered with use on account of the strain introduced by the differential expansion of quartz and platinum. For most purposes this objection is of small consequence.

The relation between the temperature $t°$ C and the resistance r_t of the platinum resistance thermometer is of the following form where a, b, and c are constants determinable by three standardization points:

$$r_t = a + bt + ct^2.$$

If only two lead wires are carried from the thermometer coil to the indicator, the resistance measured is the sum of the resistance of the coil, the platinum or gold lead wires to the head of the thermometer, and the copper lead wires from this point to the indicator. The resistance of the platinum or gold lead wires will depend upon the form of temperature gradient along the thermometer from the bulb to the head and upon the depth of immersion. Hence this variable resistance is introduced into the temperature measurement, and changes in the resistance of the lead wires will be interpreted as changes in the temperature of the thermometer coil.

There are three general methods for minimizing or eliminating the effect of variation in lead resistance.

(1) *Use of a Coil of High Resistance.*—The resistance of the lead wires from the coil to the thermometer head and the variation in resistance of the copper lead wires are small compared to a 50-ohm platinum coil. Hence such variations in resistance as are likely to occur do not introduce serious error. Thus, only two lead wires are necessary, from the thermometer to the indicating instrument.

(2) *Compensating Lead Wires of the Callendar or Siemens Form.*—Both of these methods of compensation are applicable to measurements made with the Wheatstone bridge. The Callendar form of compensation requires four lead wires. Two of the lead wires joined to the ends of the coil are connected in one arm of the bridge, and the other two lead wires, which are "dummy" leads formed of a single loop of wire extending to the top of the

coil, are connected in the corresponding arm of the bridge. The two sets of lead wires are alike, so that variations in temperature affect each set similarly. The Siemens method of compensation described below requires three leads and is more often employed industrially in this country.

(3) *Potential Terminals.*—The use of potential terminals is necessary when the resistance of the thermometer is made very low or when the precision required is such as to make variations in difference of lead resistances inadmissible.

The general methods for measuring resistance include the Wheatstone bridge, the Thomson bridge, potentiometer, and differential galvanometer. The Wheatstone bridge is used the most extensively for industrial installations.

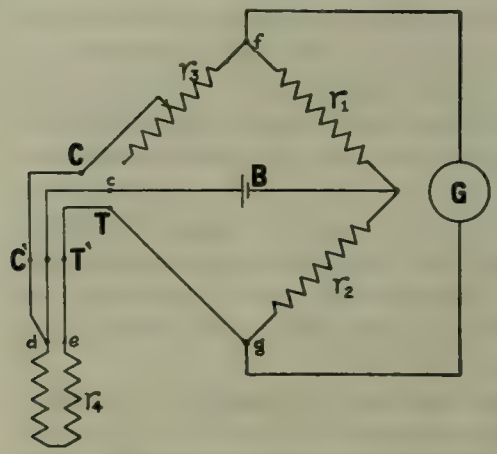


FIG. 74.—*Wheatstone bridge and thermometer with Siemens 3-lead compensation*

1. THREE-LEAD WHEATSTONE BRIDGE METHOD

Fig. 74 illustrates the wiring diagram for a simple Wheatstone bridge and thermometer with the Siemens three-lead compensation.

The platinum or gold lead wires $C'd$ and $T'e$ in the thermometer are constructed of as nearly the same resistance as possible, and the copper lead wires CC' and TT' must also be of equal resistance. The battery B is connected between the ratio arms r_1 and r_2 of the bridge and to the compensating lead wire cd. A sensitive galvanometer G is connected to the points f and g, as illustrated. By changing r_3 the bridge is balanced until the galvanometer shows zero deflection. Whence from the principle of the balanced Wheatstone bridge we obtain the following relation:

$$\frac{r_3+Cd}{r_1}=\frac{r_4+Te}{r_2}.$$

If r_1 is constructed equal to r_2 then since $Cd = Te$, $r_3 = r_4$. Hence the measured resistance is independent of the resistance of the lead wires.

2. FOUR-LEAD WHEATSTONE BRIDGE METHOD

Fig. 75 illustrates the wiring diagram for a simple Wheatstone bridge and thermometer of the four-lead potential terminal type. This method is used mainly for precision measurements in the laboratory. The battery B is first connected to c. As before, when the bridge is balanced we obtain:

$$r_3 + Cd = r_4 + Te.$$

The battery is then connected to the terminal t and the terminals T and C are interchanged by means of a commutating switch. We accordingly have for a balance: $r'_3 + Te = r_4 + Cd$. Whence upon combining these two equations:

$$r_4 = \frac{r_3 + r'_3}{2}.$$

Thus the measured resistance of the coil is independent of the resistance of the lead wires, and any error which would result from inequality of the lead resistances Cd and Te in the ordinary three-lead thermometer is eliminated.

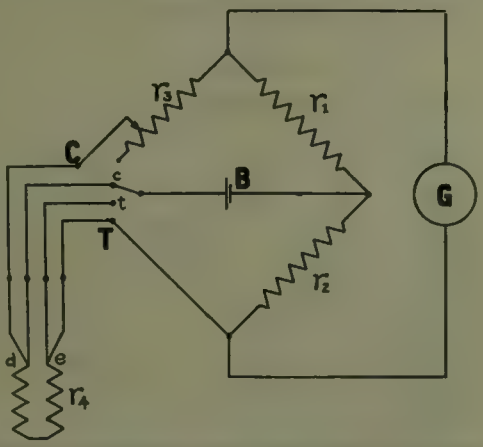


FIG. 75.—*Wheatstone bridge and thermometer with 4-lead compensation*

The diagrams of Figs. 74 and 75 are schematic and show a moving contact in the bridge arm r_3. Since resistance thermometers are nearly always made of comparatively low resistance, the variations of such a contact resistance would usually introduce serious errors. In practice the simple circuits shown in Figs. 74 and 75 are rarely used, but various modifications of the circuits are made for the purpose of avoiding the introduction of a contact resistance into the arm r_3. (See references quoted elsewhere.)

In the unbalanced bridge shown in Fig. 77, or in a bridge arranged to be connected to any of a number of resistance bulbs, a superior type of switch should be employed. If a mercury switch is not used, the switch contacts should be kept clean and tight.

3. FOUR-LEAD POTENTIOMETRIC METHOD

Fig. 76 illustrates the wiring diagram for a four-lead thermometer used with a potentiometer. The current, adjusted to a suitable value by the rheostat r_1, flows through a resistance standard R and the thermometer coil r_4. The potential drops across R and r_4 are measured by the potentiometer. Whence from Ohm's law, $\frac{e}{R}=\frac{e'}{r_4}$ where e and e' are the potential drops across R and r_4, respectively. Thus, knowing e, e', and R, the coil resistance r_4 is readily obtained. The resistance R may be made adjustable, so that by varying R until $e=e'$ the dial setting gives directly the value of $r_4=R$. This method is useful in precise laboratory work.

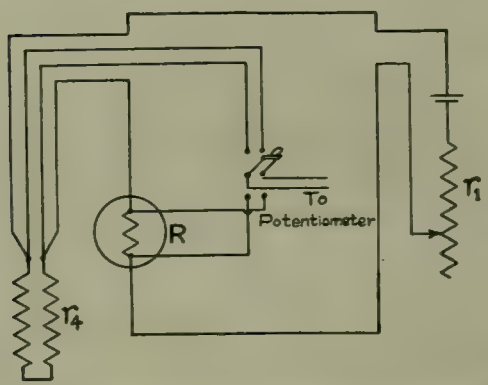


FIG. 76.—*4-lead thermometer used with potentiometer*

4. DEFLECTION-GALVANOMETER METHOD

The bridge methods so far described require the adjustment of the bridge until the galvanometer shows zero deflection. The temperature is then read from the setting of the dial or slide wire. It is possible, however, to use an unbalanced bridge with all resistances fixed, except that of the thermometer, which depends upon its temperature, and the temperature is obtained from the scale reading of the galvanometer. Fig. 77 shows the wiring diagram for an unbalanced Wheatstone bridge. With the switch S thrown to the right we have the simple Wheatstone bridge, except that the resistances r_1, r_2, and r_3 are fixed. At a certain temperature of the thermometer—for example, 10° C—its resistance is such that the bridge is balanced and $\frac{r_3}{r_1}=\frac{r_4}{r_2}$. If the temperature of the thermometer increases, its resistance increases and the bridge is no longer balanced. Thus a current flows through the galvanometer, producing a deflection. In general, the deflection of the galvanometer depends upon two factors—the temperature of the thermometer and the current flowing from the battery B. This current is adjusted to a definite value in the following manner. The switch S is thrown to the left, thus replacing the thermometer by a fixed

resistance of such magnitude that a suitable value of the current from B deflects the galvanometer to its full scale deflection or to a normal point marked on the scale. The resistance R is adjusted until the galvanometer deflects the proper amount. The switch is then thrown to the right, and, since the factor of variable battery emf or current is eliminated by this preliminary setting, the scale of the galvanometer may be empirically graduated to read directly the temperature of the thermometer. Thus no further adjustments are necessary as long as this current remains at the proper value, and any temperature is readily observed without the necessity for balancing required in the ordinary bridge. It is possible to have two or more temperature scales on the galvanometer. When two scales are provided—as, for example, 0° to 300° C and 300° C to 700° C—one of the arms of the bridge is made adjustable to either of two resistances, one giving zero deflection for a thermometer temperature of 0° C and the other zero deflection for a temperature of 300° C.

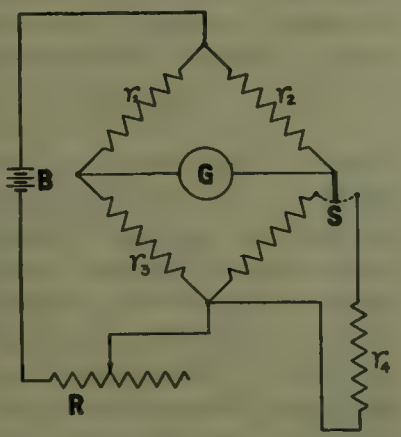


FIG. 77.—*Unbalanced Wheatstone bridge.*

5. SUMMARY STATEMENT ON RESISTANCE THERMOMETRY

Although the resistance thermometer is of the greatest importance in the laboratory for work of extremely high precision, its importance for industrial processes at high temperatures is waning. To obtain the precision of which the instrument is capable requires many precautions easily overlooked. Bad contacts in the line, a broken strand in a flexible cable, etc., may cause large errors. The thermometer is very fragile, and its calibration may be altered by severe handling or by the slightest contamination or by volatilization of the platinum wire forming the coil. The range is confined usually below about 800° C. In the early development of pyrometry, before satisfactory galvanometers were devised for thermocouples, the resistance thermometer afforded about the only accurate means of temperature measurement, but with the highly sensitive and reliable thermocouple indicators and portable potentiometers, and with the reproducible Le Chatelier couple now obtainable, there are few industrial processes carried out above

300° C where a thermoelectric installation is not to be preferred. When very small temperature changes or intervals must be measured with high precision, the resistance thermometer may be employed effectually. Also, in certain processes where an integrated or average temperature over the entire bulb is required, the resistance thermometer is highly desirable. A homogeneous thermocouple indicates the temperature of its hot junction, while a resistance thermometer indicates the average temperature of the entire coil or bulb, which may be constructed as long as desired. There are no cold-junction corrections for the resistance thermometer—a decided advantage over the thermocouple, but one which is offset by the disadvantages mentioned.

VIII. RECORDING PYROMETRY

One of the fundamental principles of efficiency is the use of adequate and permanent records. The rapid increase in the manufacture and use of recording pyrometers is a proof of the appreciation of efficiency principles on the part of the manufacturers engaged in the various technical industries. Where recording pyrometers are not employed in an industry in which temperature measurements are necessary, one will generally find that a printed form is used upon which is written, periodically, the temperatures measured by an indicating instrument. It is no serious condemnation of the workman to state that such records are often "doctored," but it is rather a reflection upon the executive who places such temptation in the way of a workman. One of the well-known "tricks of the trade" is to "force" a furnace so that it will be at the proper temperature during the periodic trips of the foreman or other official.

Indicating instruments and recorders may be used together to great advantage. The recorder furnishes a printed record and a check upon the operator of the furnace, and a record of value in correlating properties of the finished product with the heat treatment. The indicator should be of assistance to the operator in controlling the furnace or oven.

Modern practice requires a temperature-recording instrument that is as simple as possible, rugged, reliable, and sufficiently accurate. Of these qualifications reliability is paramount, particularly in cases where the recorder is used for controlling the temperature automatically. By a reliable instrument is meant one that will run continuously with little attention and with a con-

sistent degree of accuracy; that is, if it is in error by 5 per cent one day it must be in error by a like amount on any other day, and not by 2 per cent or 10 per cent. The earlier forms of recorder were complex and delicate, or mechanically unsatisfactory and inaccurate, and required considerable attention to keep them in operation. Within the past few years, however, the development in the manufacture of temperature recording devices has been highly satisfactory, and many excellent instruments are now available.

The pyrometers that can be made to record automatically fall under the following classifications: (1) Gas, saturated vapor, and liquid thermometers; (2) resistance thermometers; (3) thermoelectric pyrometers; (4) radiation pyrometers.

Of these four types, the thermoelectric pyrometer recorder has the greatest applicability, especially for the higher temperatures at which the first two named are not suitable. The constant-volume industrial gas thermometer is successful up to about 400° C. The resistance thermometer is capable of very high accuracy up to 1000° C. At such high temperatures, however, thermocouples are more serviceable, since deterioration of the wire from continual heating does not so seriously alter the emf developed by a couple as it does the resistance of a resistance thermometer. Base-metal couples serve satisfactorily up to 1100° C, and platinum platinum-rhodium couples up to 1500° C, although above 1400° C it becomes very difficult to protect the couple from contamination by the furnace gases and vapors. Radiation pyrometers are useful at the highest attainable temperatures, but processes in which temperatures greater than 1600° C are used are not, in general, subjected to very precise temperature control.

1. FORMS OF TEMPERATURE RECORDS

The most usual form of temperature record is that in which temperature appears as one coordinate and time as the other coordinate. The temperature-time curve drawn on such a record has been called an autographic record. This type of curve is the most easily obtained mechanically and is valuable as a continuous record of the temperature of a furnace over a prolonged run. It is also occasionally used to detect transformation points in steel, which appear as flexures or indentations on the plot when the furnace containing the sample is uniformly heated or cooled.

For this latter work, however, the "differential-temperature" curve recorder is especially adapted and will be described later. Other types of curves obtained with special recorders, and used mainly for laboratory work, are the temperature-rate curve, the inverse-rate curve, and the derived-differential curve. The various special methods have been discussed by Burgess[18] and will not be considered in the present paper.

2. GENERAL TYPES OF THERMOCOUPLE RECORDERS FOR TEMPERATURE-TIME CURVES

A recorder for obtaining a temperature-time curve consists essentially of an electrical measuring instrument with a mechanism for periodically recording its indications upon a chart that moves with a uniform speed. As in the case of simple indicators, there are two general types of recorder, one operating on the galvanometric principle and one operating on the potentiometric principle. Also, as in the case of indicating instruments, the potentiometric principle, while somewhat more complicated, has the especially desirable feature that its indications are independent of the thermocouple resistance. The readings of a recorder operating on the galvanometric principle depend on the variations in the resistance of the external electric circuit, although the effect of these variations can be reduced by using an instrument of high internal resistance or, with less satisfaction, by keeping the resistance of the external circuit very low. In many of the recorders now obtainable the resistance is sufficiently high that these effects become of little practical importance, or the instrument may be equipped with one of the compensation devices described elsewhere.

3. RECORD CHARTS

There are three types of record paper in general use—the roll charts and drum charts shown in Fig. 78 and the disk or circular charts shown in Fig. 79. The roll chart may contain enough paper to last a month or more, while usually the drum or circular charts are renewed every 24 hours. For single-point recorders all of these forms are employed; but, with one or two exceptions, multiple-point recorders use record paper in the roll form.

Upon circular charts, the lines of equal temperature (time coordinates) are represented by concentric circles, and lines of equal time (temperature coordinates) by arcs following the course

[18] B. S. Sci. Papers, No. 99.

of the galvanometer pointer. The distorted appearance of such a record is at first somewhat confusing, but, with practice, the record is easily read even when the complete curve is allowed to

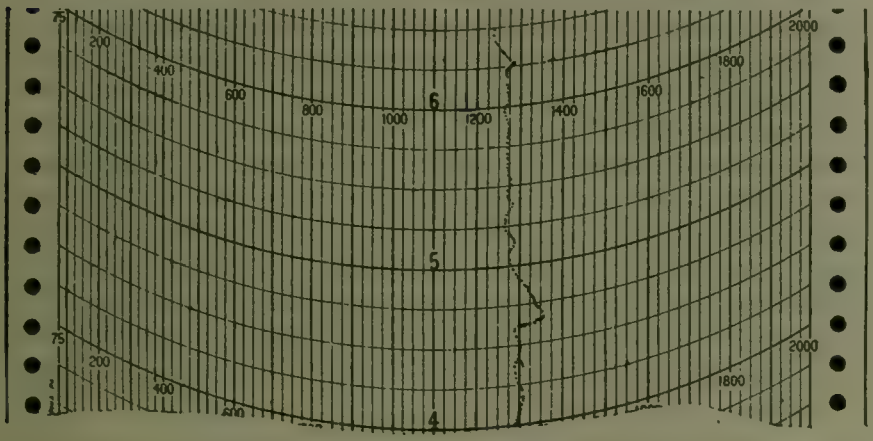


FIG. 78.—*Roll or drum chart*

extend around the chart for several revolutions. If an extended and open-temperature scale is required, the disk record becomes somewhat unsatisfactory on account of its size, since the diameter of the circular sheet must be more than twice the width of the temperature scale. The necessity of opening the recorder case, usually every 24 hours, to mount a new record sheet means greater liability for an accumulation of dust.

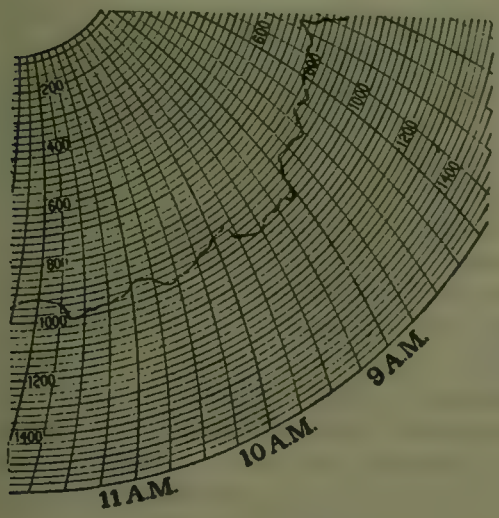


FIG. 79.—*Disk chart*

In filing records for future reference the circular and drum type one-day records offer some advantage over the continuous-roll records, although the latter may be cut to any convenient length when removed from the recorder. With the drum and roll records the coordinates can be made rectangular. In some cases, however, the temperature coordinates are parts of a circle, as shown in Fig. 78, where the radius of the circle is the

length of the galvanometer needle. The roll record is particularly adapted to multiple-point recording and for adjusting to very slow or very rapid record speeds.

The method by which the printing of the record is accomplished will be described in the discussion of the various instruments. The record may be obtained by pen and ink, stylus and inked ribbon, inked thread, carbon paper, or coated paper; by puncturing the record paper by means of an electric spark; or by stamping upon the record sheet some imprint from a stencil. The difficulty in making several distinct records upon the same chart, required by a multiple-point recorder, has been met in various ways, but there is still opportunity for development and improvement in this regard. The advantage of a multiple-point recorder for extensive installations is obvious in that for a small additional cost one instrument will do the work of several single-point recorders.

4. THERMOCOUPLE RECORDERS OPERATING ON THE GALVANOMETRIC PRINCIPLE

The recording millivoltmeter, or galvanometer, may employ the same galvanometer system used in the portable indicating instruments. A more rugged instrument is desired, however; this ruggedness is obtained by increasing the strength of the springs, boom, coil, and jewel bearings of a pivot instrument, etc. To compensate for the resulting decrease in sensitivity, the density of magnetic flux through the coil may be increased, the number of turns of wire on the moving coil may be increased, and the so-called swamping resistance in series with the moving coil may be decreased. Most indicators have a single magnet, while recorders have as many as four or six magnets. A portable instrument would become too heavy with so many magnets. In general, the resistance of a recorder is less than that of an indicator of the same type. On account of the very small emfs developed by thermocouples, and on account of the necessity of using a comparatively high resistance in series with the galvanometer coil to minimize the effect of a varying resistance, the torque that can be produced on the moving coil is small. Pivots, suspensions, etc., must be very carefully made, and the moving coil must be accurately balanced and so mounted that it swings perfectly free.

The galvanometer pointer can not be used to trace a legible record directly, since the friction between the paper and the pointer would entirely alter the readings. One common method

for obtaining the record is illustrated by the Siemens & Halske recorder, shown in Fig. 80. The paper is unwound by clockwork at a uniform speed from a roll. An inked ribbon lies below the paper and above a metal plate. At periodic intervals the chopper bar *B* falls, pressing a stylus on the end of the galvanometer boom *N* into contact with the paper and against the ribbon and metal plate underneath. This makes a small dot on the underside of the thin record paper, which shows through from the top, as illustrated. The paper is ruled with the proper time and temperature coordinates, and the row of dots obtained by continuous operation constitutes the required temperature-time curve. The maximum frequency with which the dots can be recorded depends on the natural period of vibration of the moving coil. In general practice, the dots appear at intervals of 10 to 30 seconds.

This principle is employed in many instruments of American make. The chopper bar may be operated by an electric motor, clockwork, or electromagnet, and the design must be such that the galvanometer boom swings clear of the bar, between the intervals of depression, and such that the depression of the bar against the boom in no way damages the coil mounting. These conditions may be satisfied in pivot, suspension, and combination pivot-suspension systems.

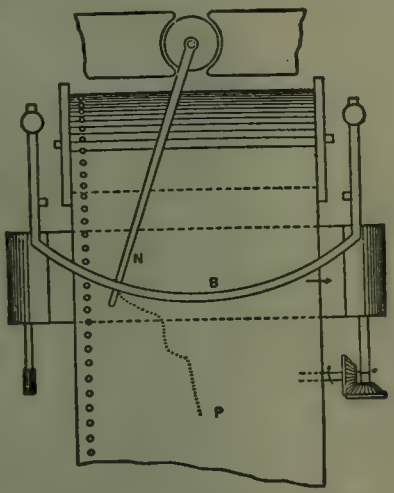


FIG. 80.—*Principle of autographic recorder*

Recorders may be classified according to the type of support employed in mounting the moving coil. The double-pivot support is the most common and was developed for its greater ruggedness and the constancy of sensitivity obtained. The outstanding fault of the double-pivot instruments is the tendency to stick and the failure of the manufacturers to make them sufficiently dust proof. An indicator of the double-pivot type may have its moving coil so mounted that the pivots do not center in the jewel bearings, but roll around in a small arc over the cup-shaped surface of the bearings. This reduces the friction considerably, but the method is not applicable for recorders in which the pointer is periodically struck by a bar with sufficient force to move the pivots in their bearings.

Unipivot-suspension instruments are not common and are not widely known. The suspension can be made very long, and hence comparatively heavy and strong. By crimping the suspension or by supporting it on a spring, a definite fraction of the weight of the moving coil may be taken from the pivot. The coil requires clamping during shipment and must be mounted in such a way that it will always seat properly if jarred out of the bearing. There is, of course, very little danger of the suspension breaking, and this type of construction can be followed in making an instrument of good constancy and sensitivity at relatively low cost.

FIG. 81.—*Types of circular-chart galvanometric recorders*

The instrument shown is made either for single or multiple records

All the advantages of the foregoing types of galvanometer construction are found in the double suspension, and with the latter a still greater sensitivity is possible. The coil is mounted so that jarring can move it a very small distance, and the spring supports of the suspensions are so made as to allow this amount of motion without undue tension. The coil is usually short-circuited when the instrument is not in use.

Recorders may also be classified according to their motive power. For most precise movement of the chart a clock must be used either alone or in conjunction with an electromagnet, which

may be applied to do most of the work. The clock alone must be large, and have plenty of power, particularly for multiple-point recorders. The commutators of multiple-point recorders must operate with considerable friction; this is best overcome by means of an electromagnet operated by the clock. The magnet is also used in some instruments to operate the chopper bar. By having the magnet lift a weight with each stroke, it can be used to furnish power for driving the chart, as is done in one of the instru-

FIG. 82.—*Types of circular-chart, single-point galvanometric recorders*

ments described below. A motor may be used for the driving power, and by attaching a governor very satisfactory control of the chart motion is obtained. The motor requires somewhat more attention than a clock, but in some recorders it is indispensable.

(*a*) INDUSTRIAL TYPES OF RECORDERS.—Foreign designs will not be described, for the writers believe that no foreign-made recorders compare favorably with the American makes in simplicity of construction, ruggedness, and general applicability for

industrial use. The American manufacturers first in the field supplied a need by making a fairly cheap form of single-point recorder simply made and sufficiently accurate to be well worth its use. This was done at a time when the imported instruments were expensive, complicated, and accurate only when cared for by an expert.

(1) *The Circular-Chart Single-Point Recorder.*—The circular-chart single-point recorders are made by a number of American firms. Figs. 81 to 83 illustrate the various forms of this type of instrument. With the exception of the Cleveland recorder, Type *c*, Fig. 81, this type is not made for multiple records. Attention might be called to the fact that the time coordinates of the circular-chart recorders are relatively short and thus these charts are unsuited to the measurement of rapidly changing temperatures. They are best suited for furnace or oven operation where a fixed temperature is maintained or desired or a slow rise or fall as met with in the ceramic industry and in annealing ovens.

Fig. 83 shows the construction of the Brown recorder. The millivoltmeter is mounted on the door so that charts may be renewed without danger of injuring the millivoltmeter pointer. The presser frame is also mounted on the door and is operated by the reciprocating arm shown at the upper left-hand side of the chart. This arm is slowly drawn back by the clock (hidden by chart) against the tension of a spring. Its release throws the presser frame against the pointer and a dot is registered on the chart. In Fig. 83 is seen a metal guide extending across the paper just below the point where the record is made. A heavy paper having a slot along this guide is placed underneath the thin paper chart and over the carbon paper so that in changing records the paper is not easily soiled. The small spool seen in the upper right-hand corner is a resistance in series with the moving coil of the meter. One of its purposes is for calibrating the instrument or changing the temperature range—e. g. to change a range 0 to 1000° to 0 to 1500° the total resistance of the instrument would be increased by approximately 50 per cent. This should not be done with low-resistance instruments without also correcting the lead resistance; that is, the correction for range should apply to the entire electric circuit.

The Hoskins millivoltmeter is unique in the fact that the meter is behind the record and the glass window closes flat against the chart, sustaining the pressure of the pointer when the latter is

raised by the presser bar. This construction gives greater convenience in changing records.

The Bristol recorder is shown in Fig. 82. The pointer has a flattened part so that when depressed by the chopper bar there

FIG. 83.—*Types of circular-chart, single-point galvanometric recorders*

is the slight rubbing action on the chart necessary with the use of smoked paper.

The Cleveland recorder shown in Fig 81 is much less simple in construction than the foregoing types. Its distinctive features are a somewhat more open scale (larger chart), a unipivot-suspension meter, and a unique method of making the record. The

pointer is not periodically depressed, but swings free with its tip close to the record. Every 30 seconds an electric spark passes from the pointer to the chart plate, puncturing the paper. The record is a series of holes with seared edges which are easily seen. There is a tendency of the spark to jump the gap at an angle, causing a slight error, which, however, is not serious. This instrument is not designed particularly for installation near the furnace, but in an office or central pyrometer station. Its high resistance makes it possible to use very long leads of small wire. The clock is electrically wound and requires no attention except regulation. To accomplish multiple recording with this instrument, the chart is divided into sectors, and, if six records are to be made, the chart moves forward slightly more than a sixth of a revolution each one-half minute. The time coordinates are very short, and the instrument is adapted in this form only to large furnaces with constant or slowly changing temperatures. It should be used combined with an indicator for controlling temperatures.

The Stupakoff recorder is the only drum chart type. This instrument is of the double-pivot type with a high resistance and ordinary scale length. The drum is rotated once in a day or a week.

(2) *Roll-Chart Recorders.*—The general advantages of the roll chart are legibility (coordinates are parallel and may be made rectangular), width of scale, and adaptability to rapidly changing temperatures, and the recording of more than one record on a single chart. With a paper speed of 1 inch an hour a 20-yard roll will last 1 month. Rolls are usually furnished in 20-yard lengths and longer.

Fig. 84 illustrates the Brown continuous recording pyrometer. The fundamental principles of its operation are the same as those of the circular-chart recorders.

Fig. 85 illustrates the Brown multiple-recording pyrometer for recording 1 to 10 temperatures. Records are obtained in five colors from a multicolored ribbon placed as shown in Fig. 80. For more than five records double dots are used. This is a method which has been found to give very easily distinguishable records. The record is made by pressing the galvanometer pointer down upon the chart which runs over a nearly sharp straight edge with the ribbon between. This results in rectangular coordinates. The length of scale is 6¼ inches (16 cm). The recording interval is 30 seconds, which gives a five-minute cycle for 10 records. The

galvanometer is of the two-pivot type with a moving coil of 50 ohms of copper and a swamping resistance of 350 ohms of manganin.

The instrument is run by electric power obtained from 110 or 220 volts alternating-current or direct-current lighting circuits or from storage batteries or dry cells. The speed is controlled by a clock automatically wound. The clock closes an electric contact in series with a solenoid magnet, which operates the depressor, and the selective switch advances the chart paper and ribbon, and is also linked to the reroll cylinder. The entire mechanism including the galvanometer is mounted on the back of the recorder case and hinged to be swung outward. The case of aluminum is designed to be dust and fume proof without an outer extra cabinet.

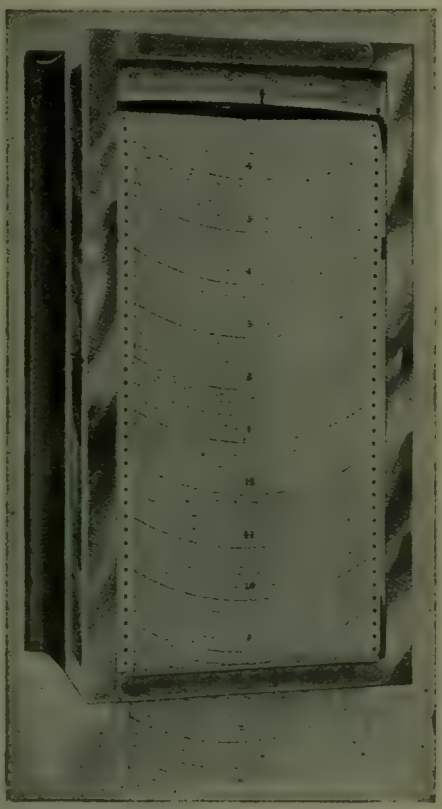


FIG. 84.—*Types of roll-chart galvanometric recorders, single-point recorder*

The Engelhard recorder which has been recently developed is of the high-resistance double-suspension type. The chart has rectangular coordinates and a graduated width of 4½ inches (11.4 cm). The resistance is upward of 700 ohms for rare-metal thermocouples.

The Hoskins multiple recording pyrometer, which has been recently developed, is shown with cover and chart removed in Fig. 86. It is made for 10 records of base-metal thermocouples. The scale is over 7 inches (17 cm) wide, the highest range instrument reading to 2500° F (1370° C). The essential features are an electric drive (*C* is a solenoid arm) and a clock operating only the electric contact device *D*. The motion of the solenoid arm raises the frame *I* (which is depressed in other instruments), changes the

commutator or rotary selective switch *H*, and moves the chart a step forward every third contact. Records are made every 20 seconds, the change from one couple to the next occurring every minute. Instead of using different colors or symbols for different couples the chart is divided by parallel lines, between any two of which only one couple records. The spaces are numbered con-

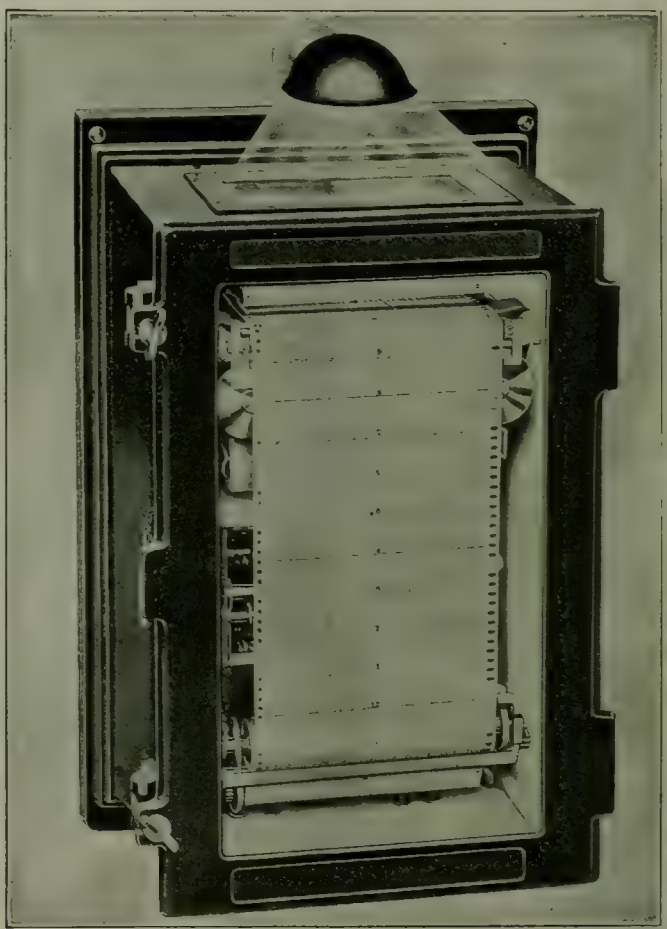


FIG. 85.—*Types of roll-chart galvanometric recorders, multiple-point recorder*

secutively and the records are distinguished by referring to the parts of the chart in which they fall. This is more clearly seen by pointing out that the selective switch is fastened directly on the shaft *G*, which holds the toothed wheels engaging perforations in the paper chart. A plug switchboard *LK* mounted within the case is so made that it is possible to arrange the couples in any

order or to put any couple on more than one number. The resistance of the millivoltmeter is 150 to 300 ohms and is intended to operate with a fixed lead resistance of 50 ohms. The high external resistance is selected to take care of compensating leads the resistance of which is about 0.13 ohm per foot. Calibration

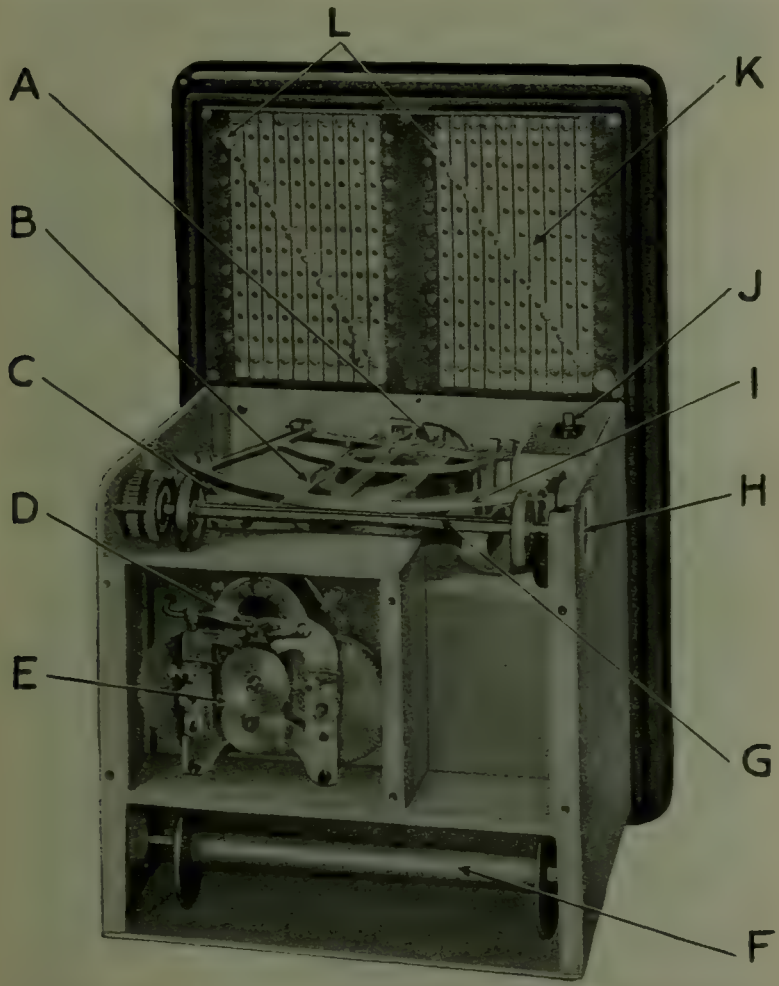


FIG. 86.—*Types of roll-chart galvanometric recorders, multiple-point recorder*

can of course be made for any external resistance met with in practice.

Fig. 87 illustrates the Taylor Instrument Co.'s thread recorder for single records. The instrument may be obtained with double scale range and the change from one range to the other is accomplished by means of a removable plug resistance located in the

lower part of the case. Using an inked thread gives rectangular coordinates. The illustration shows the ease with which replacements are made over the five small pulleys, one of which keeps the thread taut. The large clock has two parts, one of which moves the chart, while the other lifts the depressor frame. The depressor frame or bar is lifted by the clock through a pawl, which engages a slowly turning rachet. The pawl is tripped out of en-

FIG. 87.—*Types of roll-chart galvanometric recorders*

The record is obtained by pressing an inked thread against the paper

gagement, allowing the bar to fall, pressing the pointer against the thread and record paper.

Figs. 88, 89, and 90 are three views of the multipyrograph, a thread recorder for multiple records. The mechanism is built on a heavy metal frame, which slides in grooves, so that the entire works may be pulled forward for examination and cleaning, etc. The principal additions to the thread recorder are an electro-

magnet, commutating switch, and index dial showing numbers through the small opening in the left-hand box. The commutating switch is inclosed in the rectangular box below and is connected through the sprocket chain, which is turned in steps by

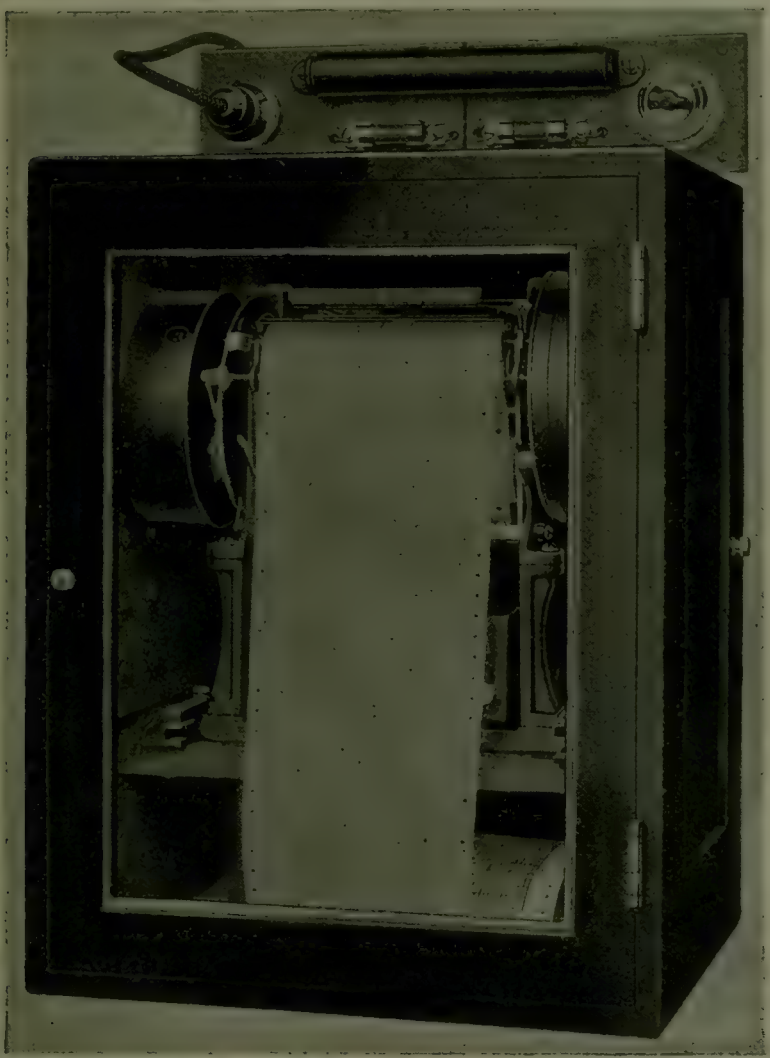


FIG. 88.—*Types of roll-chart galvanometric recorders, multiple-point recorder*

The record is made with inked threads

the electromagnet. The switch consists of a row of long, flat, phosphor-bronze springs with platinum-rhodium contacts. One spring at a time is pressed by a cam, which is properly synchronized with the numbers and colors of the threads. A wiping action

is given the contacts by an offset arrangement, assuring good contact. This principle of a wiping, or sliding, action of contacts is applied quite universally, though only in the better class of recorders are rare-metal contacts used. A high-resistance (500 ohms and more) galvanometer would render unnecessary the assured perfection of contact which is afforded by a wiping action and platinum points.

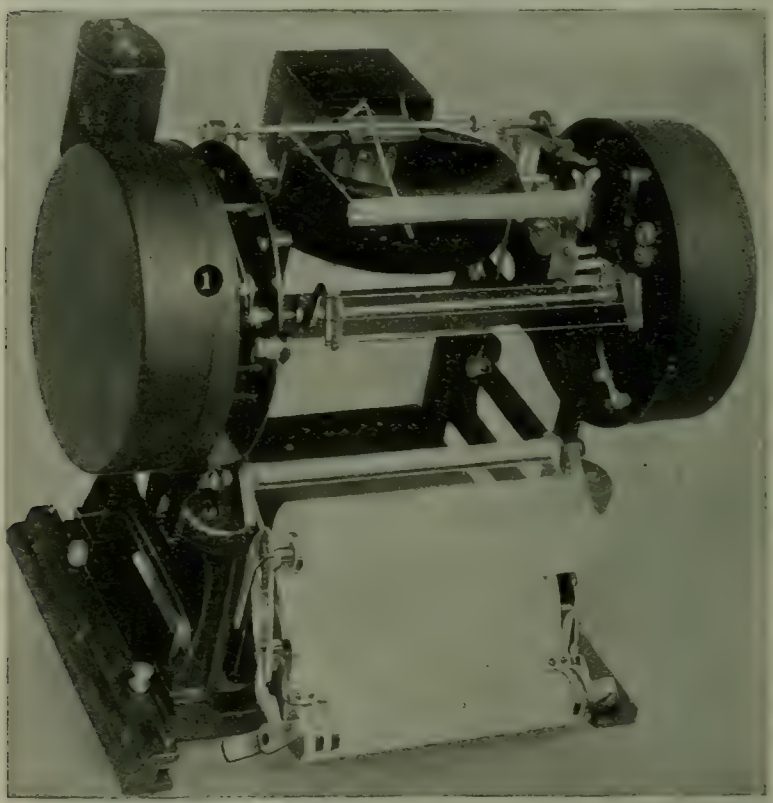


FIG. 89.—*Detail of instrument shown in Fig. 88*

The clock of this recorder does not require winding, but only provides an escapement for the gradual release of power supplied by the electromagnet. The solenoid coil raises a heavy bar, which is retained in the upper position by a pawl catching on a ratchet. The latter is allowed to turn at a uniform rate by the escapement. The motion of the magnet turns the switch cam shaft and operates the depressor arm. The chart is run through the power supplied to the ratchet by the fall of the lifted parts. Electric contact is made every one-half minute, the interval being

governed by the position of the pawl; that is, the number of ratchet teeth passed during the rise of the magnet.

Three inked threads of different colors are mounted on a frame, as shown in Fig. 89, in which the chart rolls are shown lowered into the position for replacement. The threads are brought alternately into positions governed by the commutating mechanism. Six records are obtained by changing the order of records in such a way that three records appear as a line of uniformly spaced dots and three appear as lines of dots spaced in pairs. The proper selection of numbers will usually keep records of the

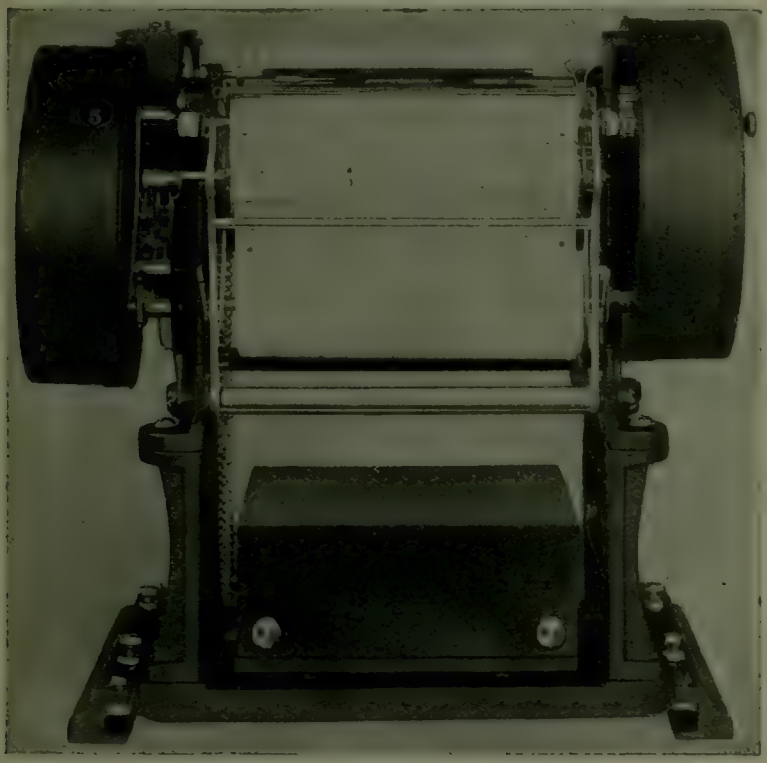


FIG. 90.—*Detail of instrument shown in Fig. 88*

same color well separated. The moving coil of the millivoltmeter is given additional protection from dust by the casing shown in Fig. 89.

To make this recorder operate on one thermocouple, the couple is connected to the six terminals in parallel. If a record of only one color is desired the threads may be replaced by three of like color. The scale of the chart is 4½ inches (11.4 cm) wide and may

be graduated in various ranges of degrees Fahrenheit and centigrade.

The Thwing recording pyrometer is illustrated in Fig. 91, which shows a type making six records with two galvanometers. Records are distinguished on this instrument by making one record of 3-minute dashes, the second of 1½-minute dashes, and the third

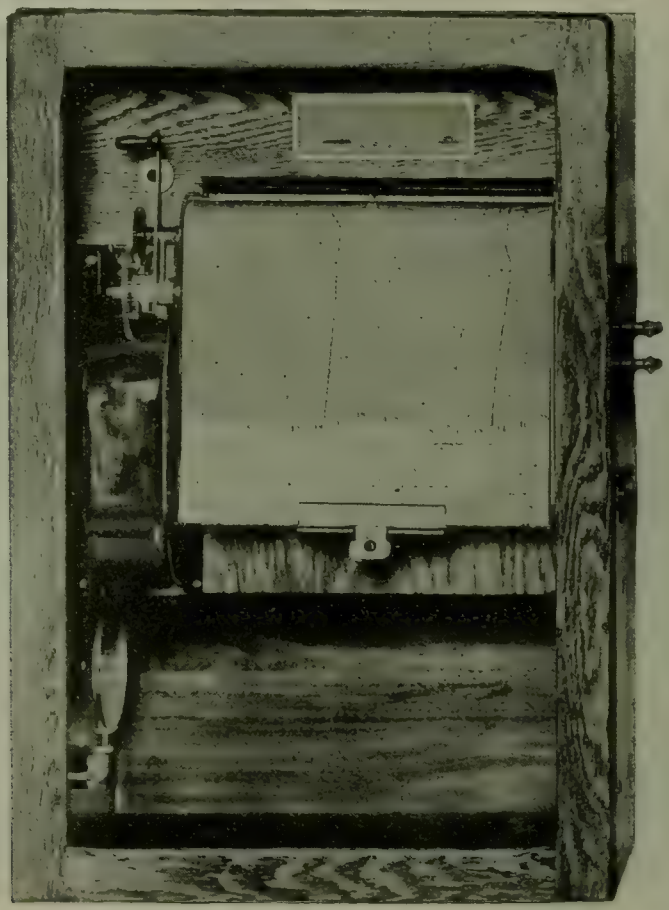


FIG. 91.—*Types of roll-chart galvanometric recorders, six-point recorder with two galvanometers*

with dots. The cycle requires 7 minutes, since both galvanometer pointers are depressed at once. After the third record the thermocouple circuit is opened, and the pointer returns to zero, where it is pressed upon an inking pad. This method of making easily distinguished records is simple and results in no confusion. The Thwing recorders are also made with three or four galvanometers

giving 9 or 12 records. The width of scale decreases with the number of records, and for 12 records, is but 2½ inches (6.3 cm) wide. The mechanism of the instrument is very simple and can be made so, even with considerable friction in operation, because the clock is very powerful and is wound once daily. The clock does all the work, turning the paper roll, depressing the needle, and closing and opening the thermocouple circuits. The commutator contacts are of silver and should give good results with a slight

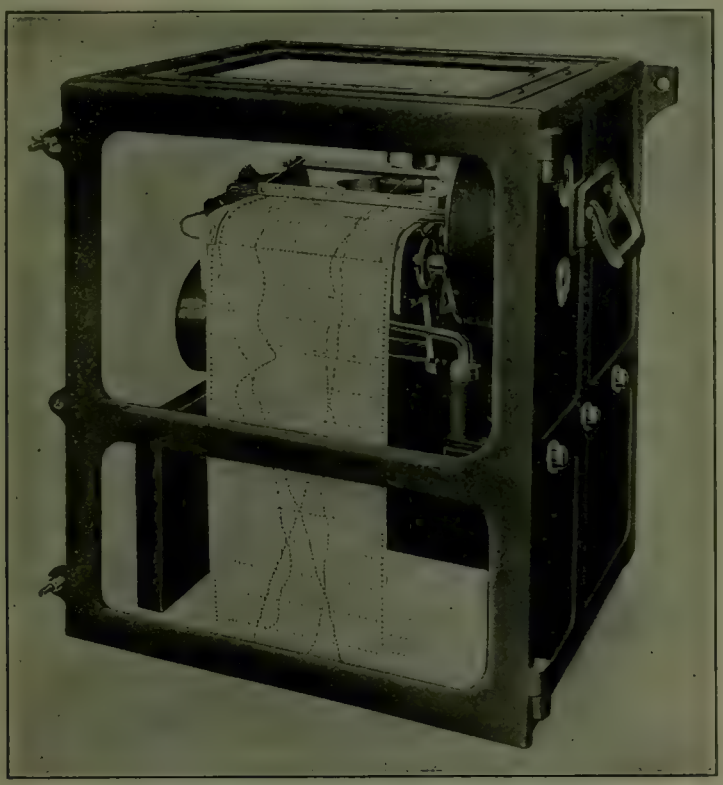


FIG. 92.—*Types of roll-chart galvanometric recorders, multiple-point recorder*

wiping action, which is obtained. This recorder is often furnished to operate on combinations of thermocouples and one or more radiation pyrometers, which is frequently very convenient. The galvanometers used are double-pivot instruments of a standard Thwing type, which has been described previously. The resistance can be made very high—in the more recent instruments as high as 20 ohms per millivolt.

The Wilson-Maeulen recorder, known as the Tapalog, is made for either single or multiple point records and is illustrated in

Figs. 92 and 93. The figures show a four-record instrument, which is the most common form. The very heavy and strong construction of this instrument is easily recognized in the illustrations. The case is heavy enameled cast iron with one-fourth inch plate-glass windows. The works are mounted on a bronze casting supported on an iron shelf. There is no need of an extra dust-proof cabinet, and this case is bolted directly to a wall.

Rectangular coordinates on the chart are obtained by pressing the galvanometer pointer against a nearly sharp, straight edge extending across and underneath the record paper. In Fig. 93

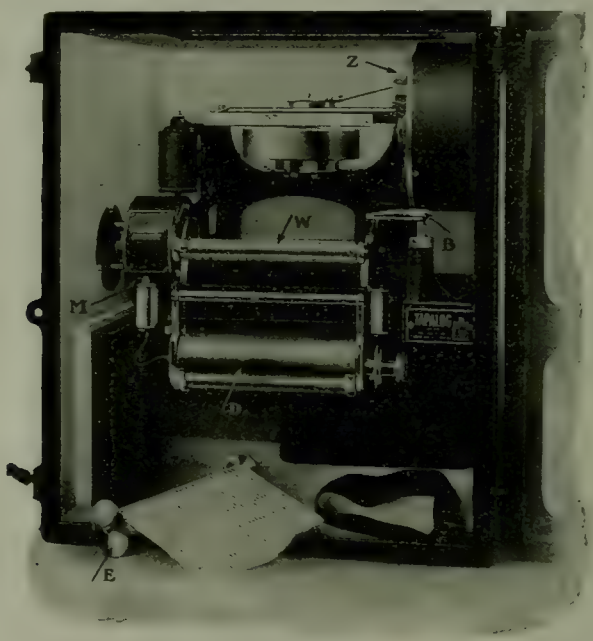


FIG. 93.—*Detail of recorder shown in Fig. 92*

the chart carriage is lowered into position for change of paper and inked ribbon, which here have been removed. The sharp, straight edge is visible midway between the sprocket roller *W* and the sprocket drum *D*. The front of the depressor frame, sometimes called the chopper bar, is straight and in line with the sharp, straight edge. The pointer is thus caught between two edges, giving a dot under the paper, which is thin and translucent. The records are distinguished by different colors. Dots are made at 10-second intervals, 8 dots to a thermocouple, and a change of couples every 80 seconds. The recorder may be made to persist in indicating the temperature of one couple by moving the lever

to "Single," which disconnects the commutator within the cylindrical box on the left.

Power is obtained from three dry cells placed within the case, and the speed of recording operations is regulated by the clock in the

FIG. 94.—*Contact mechanism of recorder shown in Fig. 92*

box at the upper right-hand side. The clock case is the background in Figs. 94 and 95. It is a powerful eight-day clock with two springs. The escapement is mechanically linked with the recorder parts through the sprocket *E*, Fig. 94, which is engaged by the

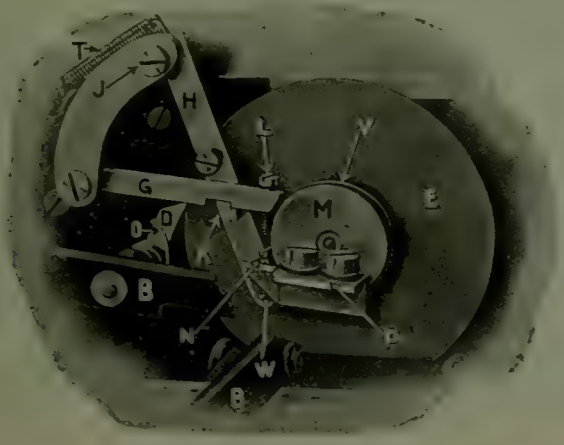


FIG. 95.—*Contact mechanism of recorder shown in Fig. 92*

pawl *D*. The chart drum is driven by the pair of gear wheels seen in the lower right-hand corner. The following description explains the operation of the contact mechanism and the method of taking the load off the clock. The depressor frame *B* is held up by

the overbalancing weight *C* and is allowed to rise slowly by the slow motion of the escapement sprocket *E*, which is engaged by the pawl *D* and its spring *O*. In Fig. 95 the corner of *B* has been cut away to show parts *P* and *W*. When electric contact (battery circuit) is made, the electromagnet forces the frame *B* down with a sharp blow. This lifts the weight *C*. The part *P*, bolted to *B*, engages the lug *W* on the arm *H*, forcing the latter to the left until it is caught by a lug entering the notch *K* in the arm *G*. As soon as the frame *B* is depressed it starts rising slowly with the motion of *E*. When the frame *B* reaches a certain point, it lifts the arm *G* (*P* strikes *L*) disengaging it from the lug on *H*, allowing *H* to be pulled into its original position by the spring *T*. The latter operation is sudden, so that the lower end of *H* strikes the spirally wound wire *V* on *M*, making electric contact in the battery circuit. Thus the cycle is complete. In this cycle, energy is intermittently stored up in the spring *T* and the weight *C*. The contact on *H* and the wire *V* are platinum-rhodium and platinum, so that good contact is always made.

Eight strokes of an electromagnet store up sufficient energy to shift the commutator. This is not shown in detail, as the parts are so small and superposed as to make it difficult to show their operation photographically. The contacts are of rare metal and a combined blow-and-wipe contact is employed.

The galvanometer of this recorder has the unipivot system described in the section on indicating instruments and may be obtained with a resistance of 450 ohms for a 55-millivolt range.

5. THERMOCOUPLE RECORDER OPERATING ON THE POTENTIOMETRIC PRINCIPLE

The principle of operation of the potentiometer circuit has been described elsewhere. The three steps in the operation—adjusting the current through *DE*, Fig. 24, connecting the thermocouple (the galvanometer is deflected), and moving the point *G* until the galvanometer deflection is zero—are done automatically in the multiple-point potentiometer recorder.

The Leeds and Northrup curve-drawing recorder is a potentiometer recorder for one couple only. The ink pen remains in contact with the paper chart at all times, and a continuous terraced curve is drawn. In this single-point recorder, the current through the slide wire is set by hand, since a commutator would be required to do this automatically, and the addition of a commutator converts the instrument into a multiple-point recorder, as illustrated

in Figs. 96 and 97. The potentiometer recorder consists essentially of a potentiometer with the mechanical device shown in Fig. 98 for automatically changing the slide-wire contact and moving the pen across the chart. It is the perfection of this simple device that has made possible the application of the potentiometric principle to the autographic recorder.

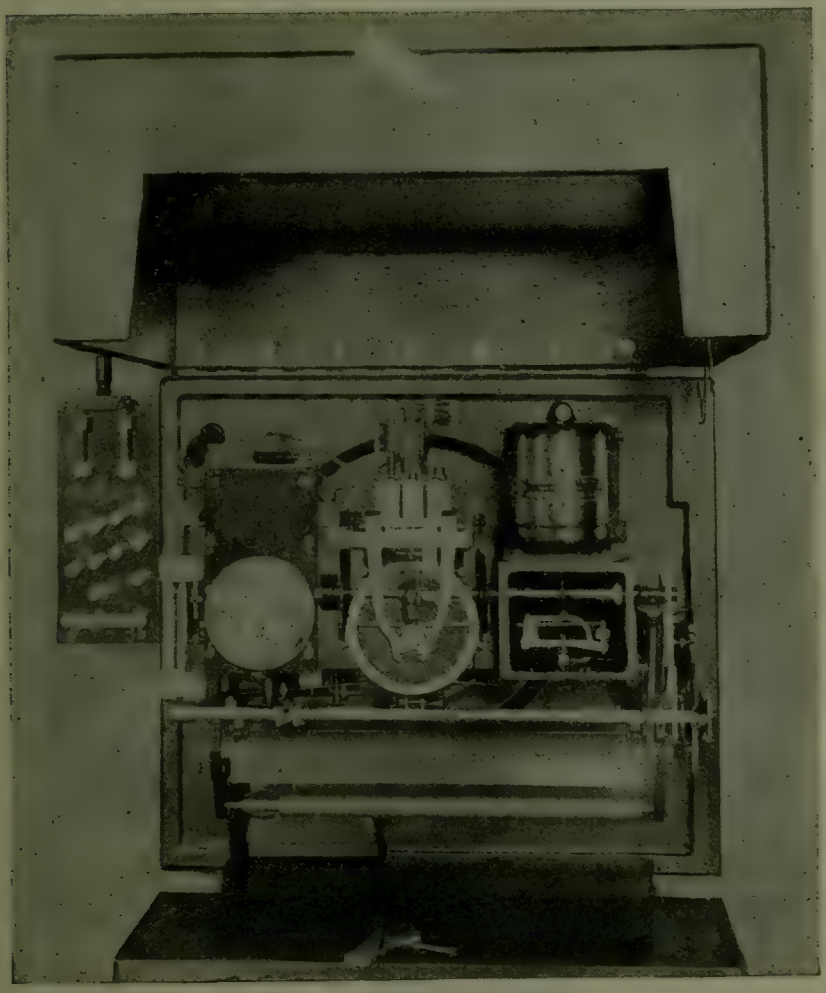


FIG. 96.—*Potentiometric recorder*

Figs. 96 and 97 show the construction of this instrument in some detail. The case consists of a cast-iron back with an oak-and-glass cover, raised in the illustrations. On the left of the case are shown the main switch, the thermocouple, and battery binding posts, and, on this particular instrument, an automatic

cold-junction compensator. On the left, within the case, is the double-pole commutator with five sectors, one of which is for the standard-cell connection. The motor is mounted above the box containing the governor, which is attached to the end of the motor shaft. The gears and cams on the right connect the main shaft with the commutator, print wheel (in place of pen), and paper chart. In Fig. 98, the moving coil of the galvanometer is marked

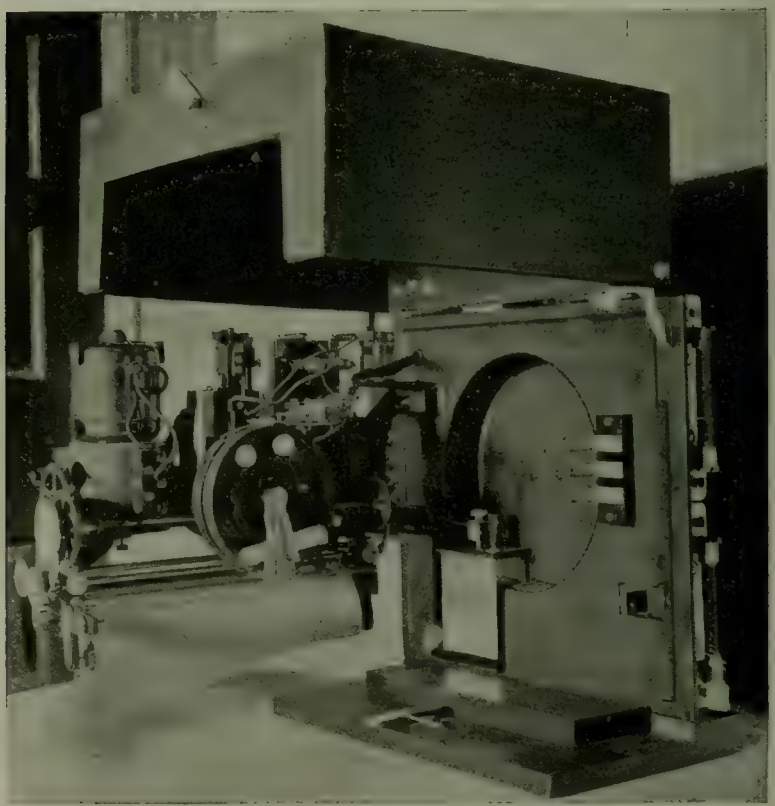


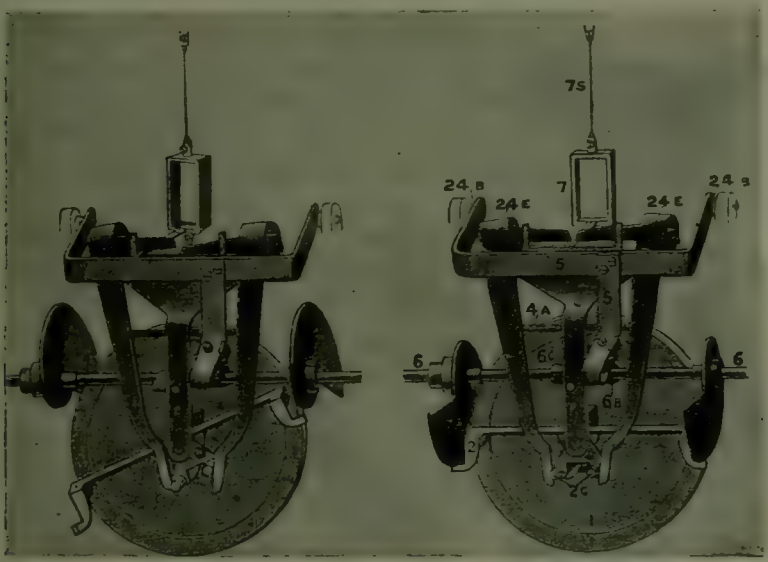
FIG. 97.—*Detail view of instrument shown in Fig. 96*

7 and this is seen in Fig. 96 between the pole pieces of the horseshoe magnet.

The chart of the Leeds and Northrup recorder is 10 inches (25 cm) wide and has rectangular coordinates. The paper has been removed in Fig. 96, showing, near the bottom of the case, the empty roll. The paper goes from this roll over the drum and out of the case through a slot in the bottom. The pen in single-point recorders, or the print wheel in the instrument illustrated, travels on a rod above the drum and is attached to a cord. In Fig. 96

this rod is turned in synchronism with the commutator, so that the proper number on the print wheel is down. The rod is kept away from the paper, against the tension of two helical springs, by arms at each end, which are pressed against the peripheries of two cams on a shaft running in the rear of the frame seen in Fig. 97, where the entire works is swung outward on a heavy cast-iron frame.

A short description of the device shown in Fig. 98 and its cycle of operations will explain how the deflection of the galvanometer results in a movement of the slide wire and pen without requiring that the galvanometer do any work. The disk 1 is mounted on



(*a*) mechanism uubalanced (*b*) mechanism balanced

FIG. 98.—*Balancing mechanism of potentiometric recorder*

the same shaft as the slide resistances *R* and *DE*, Fig. 24, which are wound on the circumferences of the disks visible in Fig. 97. The power supplied by the motor enters this mechanical system through the shaft 6 carrying the large cams 6*E* and the small cams 6*B* and 6*C*. At each revolution of the shaft 6 the cams 6*E* straighten out the arm 2, which, perchance, has been tilted a moment before, and in doing this rotate the disk 1, arm 2 being pressed at this time against the disk 1 by the spring 3. The arm 2 is pivoted on the spring 3, which is fast to the frame of the instrument. When the cams 6*E* have rotated until their longest radii are passing the extensions of arm 2, the cam 6*C* begins to

raise 3, lifting 2 away from the disk. When 2 is free, the cam 6*B* raises the rocker arm 5, which, in case the galvanometer is unbalanced, catches the pointer under one of the right-angle levers 4*L* and 4*R* pivoted at 24*E*. The lever 4*L* or 4*R* is thus made to swing the arm 2 by pressing against one of the eccentrically located lugs 2*C*. The rocker arm 5 is then immediately lowered to allow the galvanometer to swing freely. Cam 6*C* is so shaped and fixed on the shaft 6 that it will recede from the spring 3, allowing 3 to press 2 against the disk just before the cams 6*E* begin once more to straighten 2.

This mechanism, in its cycle of operations, moves the contact on the slide wire whenever the potentiometer is out of balance with the thermocouple and in so doing operates to obtain or restore the balance. The shaft 6 rotates once in about 2 seconds, which is slow enough to allow the galvanometer time to come to rest, or nearly so. This design is such that the amount of rotation of the arm 2 increases with the extent of the galvanometer deflection, since the pointer approaches the fulcrum of the lever 4*L* or 4*R* as the deflection increases. The motion of 5 is adjusted so that the rotation of 2 will correspond to a rebalancing step of the pen of three-fourths of an inch (19 mm) when the deflection is a maximum, and decreases uniformly to about one-fiftieth of an inch when the deflection is just sufficient to catch the boom under one of the right-angle levers. This gives sufficient rapidity of the various actions to take the pen the width of the scale in somewhat less than 1 minute. A record is made once a minute on the multiple-point recorders of standard design. The position of the pen, when a balance has been obtained just before each record, corresponds to a definite point on the slide wire, for the pen is fixed to a cord fast to the slide-wire disk and is wound or unwound with the rotation of the disk.

Once during a revolution of the commutator, the thermocouple is disconnected and the standard-cell connection made. At the same time the potentiometer slide wire is let loose from its shaft, and the clutch engages a second resistance *R*, Fig. 24. Movements of the disk then result in changing the resistance of the battery circuit, and the current is thus set to its proper value. The pen does not follow this adjustment, and no record is made of variations in the current. With batteries in fair condition, the current is easily maintained constant; but if there arises any doubt of this constancy, the recorder may be watched for a few

minutes, and when the standard-cell connection is made the first deflection of the galvanometer is an indication of the change in the current since the last adjustment. A short-circuiting contact on the slide wire carries the pen to zero on the chart when the battery has run down, thus providing ample warning under most circumstances.

The scale of this recorder is uniform when graduated in millivolts, and departs from uniformity for a temperature graduation according to the temperature-electromotive force relation of the thermocouple. The standard galvanometer is sufficiently sensitive to work satisfactorily with a full-scale range of 10 millivolts, which gives a very open scale, particularly for base-metal couples, when 0.1 inch (2.5 mm) of scale corresponds to 2.5° C. This recorder is used with resistance thermometers, in which case the electrical circuit takes the form of a Wheatstone bridge, and the scale can be opened until the entire range corresponds to as little as 2° C. The zero of the scale can be adjusted to correspond to any fixed electromotive force, so that the scale may be put within any range of temperature desired. The great adaptability of the instrument is readily apparent. Some of the applications will be considered in the section on temperature control.

6. TRANSFORMATION-POINT INDICATORS AND RECORDERS

Instruments for obtaining transformation or critical points in steels have been in use for a number of years and are fairly well known in the steel industries. The simplest, being also the least accurate method of measuring the temperature at critical points, is to record or plot from indicator readings the temperature-time curve of the sample of steel when placed in a furnace and heated. This method requires a very steady rate of heating, and the sensitivity is ordinarily only sufficient for high-carbon steels. It is possible, by using a potentiometer and very sensitive galvanometer and forcing the furnace to rise in temperature at a fixed rate, to obtain a temperature-time curve that will indicate plainly all the transformations. There is great difficulty in keeping the rate of heating constant, and it is not necessary to control this rate better than to keep it from changing rapidly. However, in the research laboratory, good heating-rate control, sufficient sensitivity, and an accurate method of measuring time intervals, as with a chronograph or stop watch, will give all the necessary

data, sometimes requiring for a complete interpretation the replotting of the data [19] as temperature versus inverse rate, $\frac{\Delta t}{\Delta \vartheta}$.

Burgess has described the use of a neutral body and the differential-couple arrangement (first devised by Sir Roberts-Austen) which tends to avoid to a large extent the destruction of useful data by variations in heating and cooling rates. The differential couple is mounted with one hot junction in the test piece and the other in a neutral body (one with no transformation points).

The data obtained are curves of temperature of test piece versus temperature difference between test piece and neutral body. These are mounted side by side in the furnace and must be of such size, specific heat, emissivity, etc., as to heat and cool at nearly the same rate.

The industrial laboratory requires a method of automatic, or at least semiautomatic, recording of cooling curves. Simplicity of the apparatus implies a limited scale range, incompatible with the direct ϑ versus t method, and requiring the use of the differential thermocouple. To avoid errors due to improper measurement of time intervals, the records of temperature and temperature difference must be on one chart and are best obtained from a single galvanometer connected alternately to the thermocouple in the test piece and the differential couple. A curve of temperature versus temperature difference may be obtained simply in a semiautomatic recorder using two galvanometers.

(*a*) The Brown Transformation-Point Recorder.—Fig. 84 illustrates the type of recorder applied for this purpose. The single galvanometer of this instrument has two windings of low resistance, one of which has in series with it a high resistance and is employed to measure temperatures. This coil is connected permanently to the thermocouple in the test piece. At intervals a record is made of its temperature. At alternate intervals the other coil is connected to the differential couple, and a record made of its temperature difference. This difference shows on the chart as the distance apart of the two curves of ϑ and $\vartheta - \vartheta'$. If the first coil were disconnected during the time the other is connected, $\vartheta - \vartheta'$ would be measured by the deflection from zero on the chart. But $\vartheta - \vartheta'$ may be positive or negative, and this arrangement would necessitate a reversing switch and offer no advantage. To obtain sufficient sensitivity base-metal couples

[19] G. K. Burgess, On Methods of Obtaining Cooling Curves, B. S. Sci. Papers, No. 99.

and rather large test pieces are used. The instrument will show a curve with a very marked jog at the eutectoid transformation point, and by close observation the allotropic transformation point A_3 may be detected in pure iron. There is not an excessive sensitivity, but practically the best that can be done with a double-pivot galvanometer.

(*b*) THE LEEDS & NORTHRUP TRANSFORMATION-POINT INDICATOR.—This instrument is a semiautomatic recorder giving a

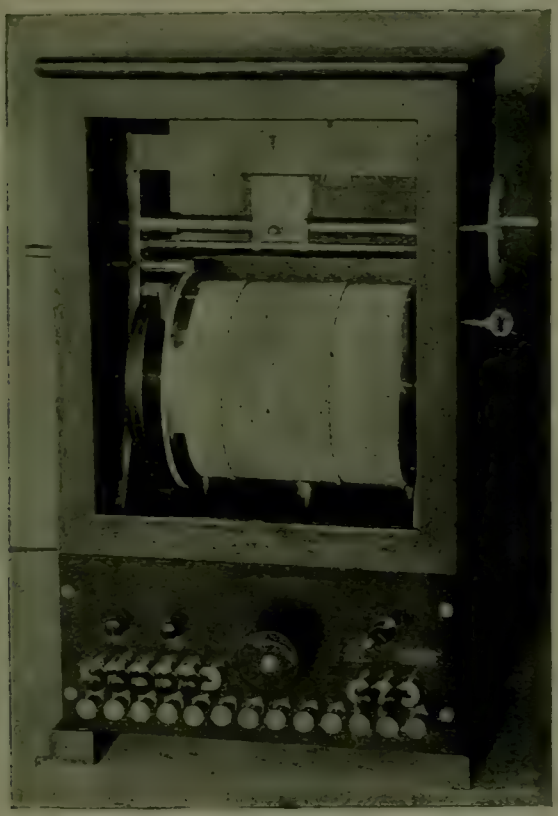


FIG. 99.—*Transformation-point indicator*

continuous curve of ϑ vs. $\vartheta - \vartheta'$. One observer is required and no time measurements are made; it is illustrated, in part, by Fig. 99. The wall-type reflecting galvanometers (two) are not shown. The complete apparatus includes the two galvanometers, furnace, rheostat, and thermocouples.

The recorder consists, in the main, of a potentiometer with a slide wire and drum chart on the same shaft. In moving the slide wire to obtain a balance of the potentiometer galvanometer,

the chart, with ordinates graduated in degrees (or millivolts), turns simultaneously and its position with reference to a pen held in contact indicates the temperature of the test piece. The pen is mounted above the chart on a carriage, which may be moved by turning a screw across the width of the chart. Upon the carriage is a ground-glass screen with a central vertical mark. A spot of light from the differential galvanometer is focused on this screen. The differential couple is connected directly to this galvanometer, and its deflections are followed by turning the pen-carriage screw, keeping the mark on the glass coincident with the light beam from the galvanometer. A separate glass scale is provided for balancing the other galvanometer. The observer has two motions to perform—viz, turning the slide wire (and drum) and turning the pen-carriage screw. The zero-temperature point is suppressed, and the end of the slide wire is usually made to correspond to 2 millivolts or the corresponding temperature. The other end of the slide wire corresponds to 10 millivolts, giving 8 millivolts, or about 800° C, over the whole chart length of 20 inches (50.8 cm). This is a sufficiently long temperature scale for all practical purposes.

In series with the differential galvanometer is a resistance used to cut down the sensitivity of the galvanometer, which will be usually deflected off the scale at a eutectoid transformation point. The sensitivity is ample for detecting all the transformation points ordinarily required in practice. The period of the galvanometer is short enough to allow the heating and cooling to be done in less than 1 hour. The curve obtained is a continuous line, slightly ragged due to manual operation, and is easily translated into metallurgical terms. It can be replotted into a curve of ϑ versus $\Delta(\vartheta-\vartheta')/\Delta\vartheta$, the so-called "derived-differential curve," due to Rosenhain,[20] especially if errors are suspected on account of considerable differences in the cooling curves of the test piece and the neutral. This curve also aids in the interpretation of results and corresponds to the inverse-rate curve when only measurements of temperature and time are made without the use of a neutral body and differential thermocouple.

[20] Observations on Recalescence Curves, Proc. Phys. Soc. Lond., 21, p. 180; 1908.

IX. HIGH-TEMPERATURE CONTROL

The meaning of temperature control may be extended to cover not only the control of temperatures, but also the control of processes through a knowledge of the temperatures involved. In this sense it has a very wide interest. Temperature control is involved in nearly all industrial processes, but the present discussion is confined to high temperatures.

Some of the factors increasing the difficulty in the regulation of furnaces, ovens, kilns, tanks, etc., are inconstancy of heat supply, variation in internal absorption or generation of heat, variation in heat lost by radiation, etc., and unsteady supply or composition of material to be heat treated. Each of these items is intimately associated with temperature and temperature variation. Hence control is best accomplished when the relation between these factors and temperature and temperature variations is understood.

In many cases the accumulated experience of years enables an operator to control a process fairly successfully without the assistance of a pyrometric installation. Frequently the process is characterized by a definite chemical or physical change in itself sufficient for control. Occasionally the operating conditions may be maintained so constant—for example, in distillation processes—that a pyrometer merely indicates a fixed temperature within the accuracy of the instrument. The operators of open-hearth furnaces secure remarkably reproducible results by visual observation of the slag and roof. In a pyrites dead-roast furnace the ore tends to become too hot and ball up, but before disastrous consequences result the operator has sufficient time to cool the furnace. Glass can be worked without a pyrometer because there is a considerable temperature range over which this is possible.

However, the operation of these and other processes may be improved by a suitable pyrometric equipment. Maximum efficiency is usually secured over a narrow range of temperature through which a process may be carried out. Thus in forcing the open-hearth furnace to maximum production the rate of heating of the charge is limited by the refractoriness of the roof brick. If the brick melts at 1710° C, the deterioration of the roof is very rapid at this temperature. But at say 1690° C, at which temperature there is no physical change to aid the operator, the life of the roof is much greater. The process, therefore, can be carried out more efficiently if this temperature is maintained by the use of an optical pyrometer. With a thermocouple installed in the hottest

bed of a pyrites furnace the temperature may be controlled below the danger point, and thus avoid the necessity of occasional drastic action to cool the furnace and lower production. Glass works best in a small range of temperature; hence, production and quality may be increased by installing pyrometers. In the lehrs, where visual estimates of temperature are impossible, the pyrometer is essential. Here it is necessary to know the highest temperature reached and also the rate of cooling of the ware. In numerous industrial processes the pyrometer is recognized as an indispensable aid to temperature control, especially in the metallurgical industries, where such close adherence to temperature specifications is required that frequently the pyrometer is taxed to furnish the required accuracy.

In order to control a furnace with a pyrometer it is often desirable to locate the instrument at the point where the largest temperature variations occur and where these variations take place the earliest. It is necessary not only to obtain correct temperature where this is desired, but also to obtain the temperature at some other point in the furnace, variations of which have a known significance and relation to proper operation. For example, a thermocouple may be so located that should a furnace tend to become too hot this tendency would be evidenced by the pyrometer indication and a cooling action could be initiated before any vital part of the furnace is overheated. The pyrometer should function in a like manner during cooling. Unfortunately in many processes the temperatures are too great for such an installation, and this fact is responsible for the slow development of automatic control at higher temperatures. Thus a thermocouple could not be employed for this purpose in steel and glass manufacture or in many ceramic processes, but possibly a radiation pyrometer could be used.

Often the position must be so chosen as to protect the couple even though the temperature indicated is not the one most desirable to know. One of the severest tests of permanently installed couples is in a pyrex glass tank. Rare-metal couples are mounted in the tank wall without extending to the inner face. Although the indicated temperatures of 1475 to 1500° C are possibly 100° below the actual temperature of the glass, the method affords a fair degree of control and is about the only means suitable for recording instruments.

The methods of rendering temperature information useful—that is, the method of indicating and recording temperatures and

their variations—are very numerous and must be adapted to special needs in a plant. The simplest plan is to mount the pyrometer indicator, preferably of the wall type, in a conspicuous place, where the operator can read it with the least trouble; special means must often be employed for making the scale sufficiently legible. The scale must be as open as possible, so that small changes of temperature may be readily detected. Some indicators are provided with two pointers, one of which can be turned to the temperature desired. This second pointer may be made a single line or a double one, the latter indicating the limits of variation allowable. In the absence of a recorder the second pointer is nearly indispensable as a means of judging rates of change. In poorly lighted rooms illuminated scales may be used. If cheap labor is employed and the operator is unable to adequately interpret the indicator reading, signaling lights or alarms may be operated either manually or automatically. In larger plants the indicators may be situated at a central station, where the pyrometer man reads them and transmits the proper signal to the operator. Colored lights are best used for this purpose, white for correct temperature, green for too low, and red for too high, and white with red or green for small departures from the correct temperature. The lighting circuit is, of course, completely separated from the pyrometer circuit. If the plant electrician is allowed to install the wiring, he must be convinced that electrical insulation and leakage assume a new significance in electrical pyrometry. The use of these colored lights or a signal system is not restricted to control at a fixed temperature; they may be employed for governing the rate of heating up a kiln, for instance. In the ceramic industries, pyrometers are particularly useful as a means of avoiding loss of time and curtailment of production on account of a nonuniform rate of firing.

The operator, in controlling a furnace to reach a desired temperature, bases his action on experience and judgment. Possibly, he turns a gas or oil valve a certain part of one turn, according to the change just observed in the condition of the furnace. Obviously the adjustment can best be made with a knowledge of the changes which have occurred over a considerable interval of time rather than by watching the change occurring from moment to moment. This is one of the most prominent advantages of a recording pyrometer; and when it is possible and the class of labor employed warrants it, the operator should be allowed the advantages of inspecting the recorder chart. It is necessary that

the record be made with a frequency greater than that of significant changes likely to occur in the furnace, particularly when a multiple recorder is used. In case a single-record recorder is used or a multiple-galvanometer recorder in which each galvanometer is always connected to one pyrometer, the position of the pointer or pen shows the change occurring. The recorder should be so constructed that the record is visible to the last minute.

The application of the multiple-point recorder affords further advantages in control. These arise from the paralleling of records on one chart, showing, for instance, the time-temperature curves of a battery of furnaces. It is also of advantage to parallel the records of two or more points in a single furnace which is being heated. Thus the rate of increase in temperature of a central portion of the furnace may be made as high as possible, while the record shows at the same time the temperature in dangerous portions of the furnace likely to be damaged by an excessive rate of firing.

When pyrometers are newly installed in a process where they have not been used before, the immediate result is usually confusion. Difficulty is encountered in correlating the indicated temperatures with other conditions, so that the operation may be at first even less efficient. Too much attention is given to temperature, and the attempt is made to operate without regard to other necessary factors, or the new knowledge is misinterpreted because of the errors of previous ideas. These usually exist and must be recognized. An operator after years of experience develops a temperature scale of his own, which, while useful to him, may differ considerably from the true temperature scale. The change in position of a damper may produce very peculiar results in the temperature of one portion of a furnace, quite contrary to the effect expected by the most experienced operator. The transition period following the adoption of a pyrometric equipment is bridged with difficulty and every effort should be made to correlate facts as they really exist, not as they have been thought to exist, with temperature.

The employment of pyrometers is generally least in those industries in which operating conditions are the worst because of the great difficulty met with in maintaining uniformity of conditions. It is particularly true of such an industry as the ceramic, in which the chemical constitution and physical condition of raw materials are of prime importance and difficult to control. The kiln operator often can not fire his kiln according to any certain

time-temperature relations because he does not know how, not because it is impossible. He will never know how so long as pyrometers are not installed and the time-temperature relations are not associated with other variables. Pyrometers are now being used in kilns in order that the rate of heating may be maintained with greater certainty and a loss of time due to too slow firing be avoided, but the finishing time is determined with Seger cones. It is hoped that the Seger cones will eventually be supplanted by even more useful pyrometric methods.

1. DEVICES USED FOR CONTROL

Control, either automatic or manual, and automatic signaling are always accomplished by the employment of a method of forcing the furnace to heat or cool between limits, at a rate and through a range depending on the process and constancy required. Ordinarily there is little difficulty in obtaining enough sensitivity of the instrument for either automatic signaling or control, except in the research-laboratory furnaces, where at times the limit of sensitivity is employed. In control of a furnace through pyrometric measurement, it is not necessary that the pyrometer be accurately calibrated. It must, however, be particularly reliable and have a consistent error, as has been stated elsewhere.

(*a*) AUTOMATIC ALARM.—An ordinary pyrometer galvanometer of low resistance may be fitted with two contacts on pivoted arms, between which the meter pointer plays. No relay is necessary for the small current and voltage required to operate a bell. The automatic alarm is seldom used in this form, since the alarm operates for too large a part of the time unless the contacts are placed wide apart, when their usefulness is much lessened. The action may be made intermittent on more complicated instruments. The alarm should properly be employed only when the departure from a certain temperature range results in real danger to life or property.

(*b*) MANUAL SIGNALING.—The development of manual signaling has taken place, for the most part, in the larger plants having extensive pyrometric installations of such scope that a central pyrometer station is necessary. Fig. 100 illustrates a form of central station developed at the Ford Motor Co. by W. G. Vesey. With such an installation only the thermocouples and signal lights are in the furnace room and the indicators in the station are connected to different couples by means of push buttons on switchboards. The more sensitive types of double-suspension or uni-

pivot-suspension millivoltmeters or thermocouple potentiometers are used and can indicate easily temperature variations of 0.2 per cent of the scale range. The substitution of manual operation for automatic is necessary to attain the closest correlation of temperature measurements and other physical conditions and the physical and chemical properties of the products.

Signaling is accomplished by colored lights, using three at each furnace. Temperatures are read at frequent intervals, determined by furnace operation and the operator's interpretation of signals. In some plants pneumatic tubes are installed between the furnace room and the station for transmitting notes concern-

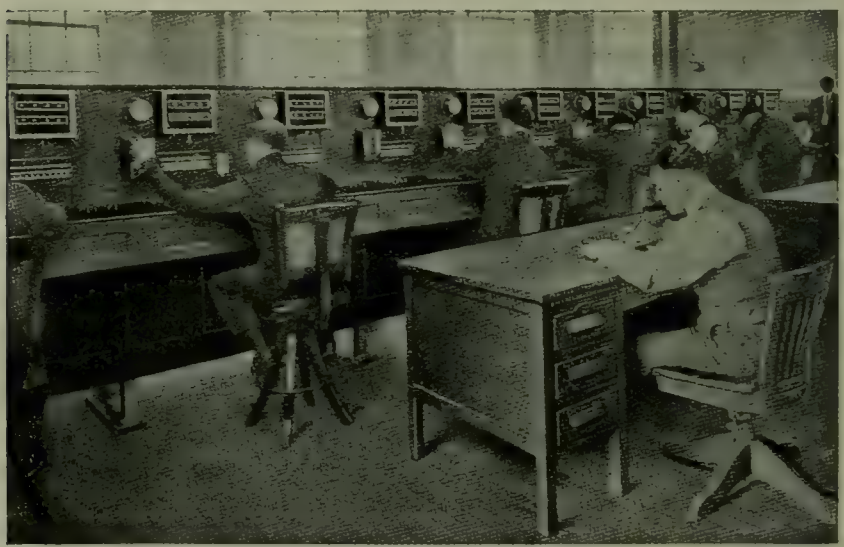


FIG. 100.—*A central pyrometer station*

ing the process or measurements. The furnace man can signal the station with the ordinary enunciator, as shown in Fig. 100, at the top of the switchboard. Such a signaling system of colored lights, enunciators, and pneumatic tubes is, of course, not peculiar to pyrometry.

(*c*) AUTOMATIC SIGNALING.—The descriptions of the various types of recorder make it clear how automatic signaling may be accomplished with pyrometer galvanometers. In a similar manner to that in which autographic records are made, a depressor bar or frame, in its rise and fall, closes either of two pairs of contacts depending on the position of the pointer. The device may be attached to an indicator, according to Fig. 101, or a recorder may

be made to do both recording and signaling. The latter arrangement has not been very successfully applied to the galvanometer type of recorder, but is easily adapted to the Leeds & Northrup potentiometer recorder. In this case two contacts move with the slide wire on its shaft and the third is stationary.

The galvanometric instrument illustrated in Fig. 101 has two platinum contacts mounted on an arm which swings across the scale to adjust the signaling range. The signaling-circuit current does not flow to the galvanometer pointer, but each platinum contact is double, one above the other, and the two are brought together by the pressure of the depressor arm transmitted through the pointer. The pointer is, in this case, a selector. The arrangement of Bristol differs from this in that a tipping device

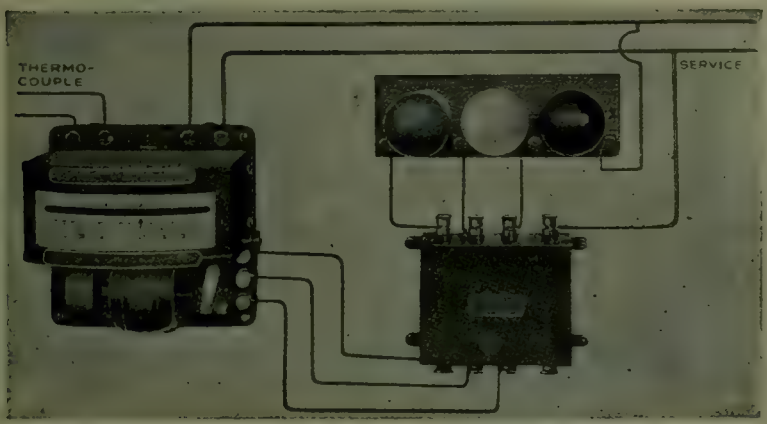


FIG. 101.—*Automatic signaling pyrometer*

restrained by springs is tipped by the pointer when the depressor arm falls in a direction determined by the pointer. The springs are arranged to snap the contact when the pressure has reached a certain value. By this means the selection can be made to take place over a smaller interval than with two contacts placed side by side and close together. The electromagnetic switches operated by closing these contacts scarcely need description. They are made applicable to any range of voltage and require a small fraction of an ampere. The circuit may include any of the well-known signaling devices, some of which have been mentioned.

Figs. 102, 103, and 104 illustrate, diagrammatically, the arrangement of the curve-drawing potentiometer recorder with signaling lamps and furnace indicator. The diagrams are self-explanatory, but the new method of supplying power to the in-

dicator may be mentioned. This is not taken from the thermocouple, but the indicator is shown in a Wheatstone bridge attached to the line, preferably 110-volt alternating current. The movement of the disk carrying the slide wire *S* and the three contacts

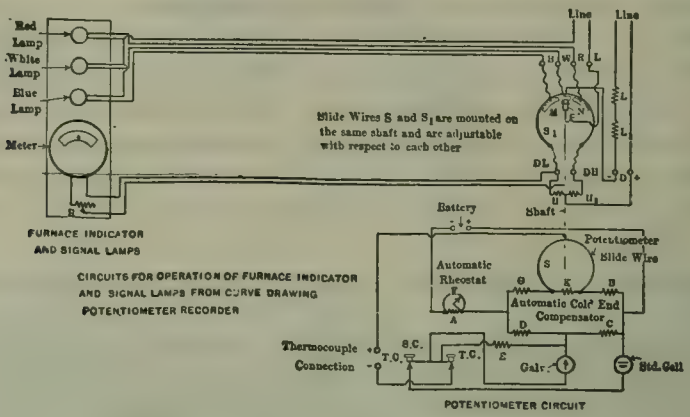


FIG. 102.—*Automatic signaling recorder, potentiometer type*

connected to the lamps results in connecting the proper lamp and in unbalancing the bridge circuit. The meter may be of high resistance and its indications are easily checked by the recorder indication and correction accomplished with the adjustable resistance in the circuit. It is only intended to show the magnitude

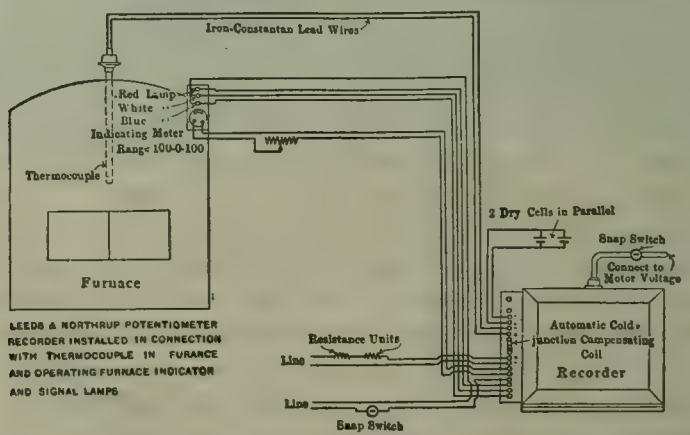


FIG. 103.—*Automatic signaling recorder, potentiometer type*

of departures from correct furnace temperatures and a large variation in line voltage would not be serious. Fig. 104 shows the use of the double recorder, or curve-drawing recorder, with a commutator for two couples, which gives the form of record

shown in Fig. 105. This instrument is also used in the automatic control arrangement shown in Fig. 108.

The advantages of automatic signaling are not always well defined, and the method has not been put to very extensive use.

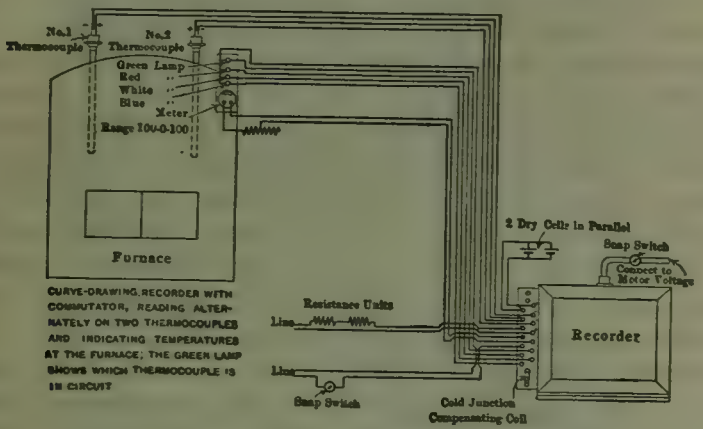


FIG. 104.—*Automatic signaling recorder, potentiometer type*

In cases where very exact temperature measurements and control are necessary and the sensitivity demanded results in an instrument which must be removed a distance from the furnace or oven, automatic signaling may be required; but in most cir-

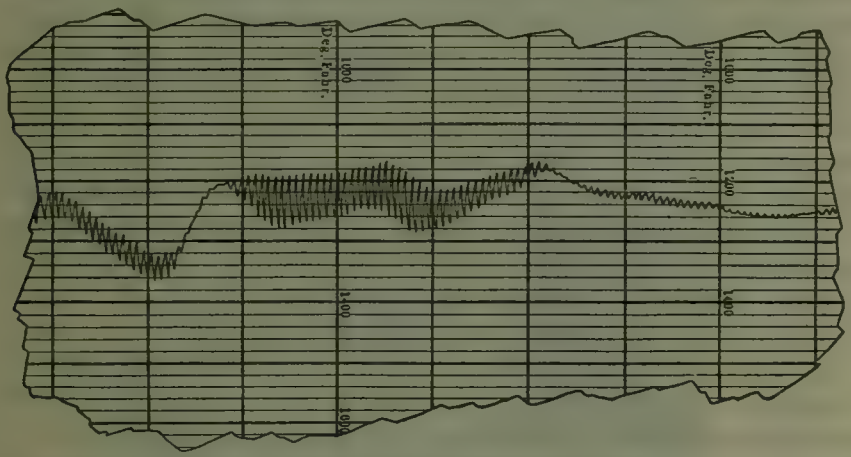


FIG. 105.—*Record of double curve drawing recorder*

cumstances the proper placing of the indicator or indicators will make automatic signaling unnecessary. To make one indicator operate many separate systems of signaling would require a complicated and cumbersome commutator. Such an arrangement

might take the place of some of the observers in the central pyrometer station of a large plant, but sensitivity and flexibility would be sacrificed.

(*d*) AUTOMATIC TEMPERATURE CONTROL.—Temperature control at low ranges is termed thermostating and is ordinarily obtained by means of the movement of bimetallic springs, or the thermal expansion of rods or fluid columns. At high temperatures these devices are utilized only with great difficulty, and thermoelectric instruments are substituted. The principle of operation is similar to that used in automatic signaling. Electromagnetic impulses arising in the pyrometer indicator or controller are utilized to operate oil or gas valves or electric switches. In case the valve or switch is large and the electromagnetic operation requires more than a few hundred watts, electric relays are incorporated between the controller and the valve.

Automatic temperature control is complicated by the interrelation of such factors as thermal lag and the magnitude of the corrective change in heat supply during each period of reversal. The sensitivity of the control instrument determines the range of temperature that must be covered by the thermocouple (or other pyrometer) during each reversal of the valve or switch. The range of temperature at the source of heat is greater than this, and the difference is determined by the type of furnace, the method of heating, and the locating of the thermocouple. For closest regulation, which is best accomplished electrically, the couple must be very near if not in contact with the heater. Considering the cycle of operations, if a furnace is cooling the current will be increased when the couple has dropped a certain amount, the heater being at a still lower temperature. The current may be increased by a single step upon reaching this point or a mechanical arrangement may be used, which will continue to increase the current so long as the couple has not reached the upper limit of temperature. The single step will ordinarily give the least overcorrection of heat supply, and the method is the most simple to apply. It will fail to give the desired result, however, if there exists a condition of wide variation in the amount of heat absorbed or lost in the furnace operation, for in this case the heating current change during a reversal must be large. A mechanism which is always increasing or decreasing the heat supply in small steps may be often advantageous and is practically indispensable in the arrangement mentioned below for automatic control of heating or cooling rates.

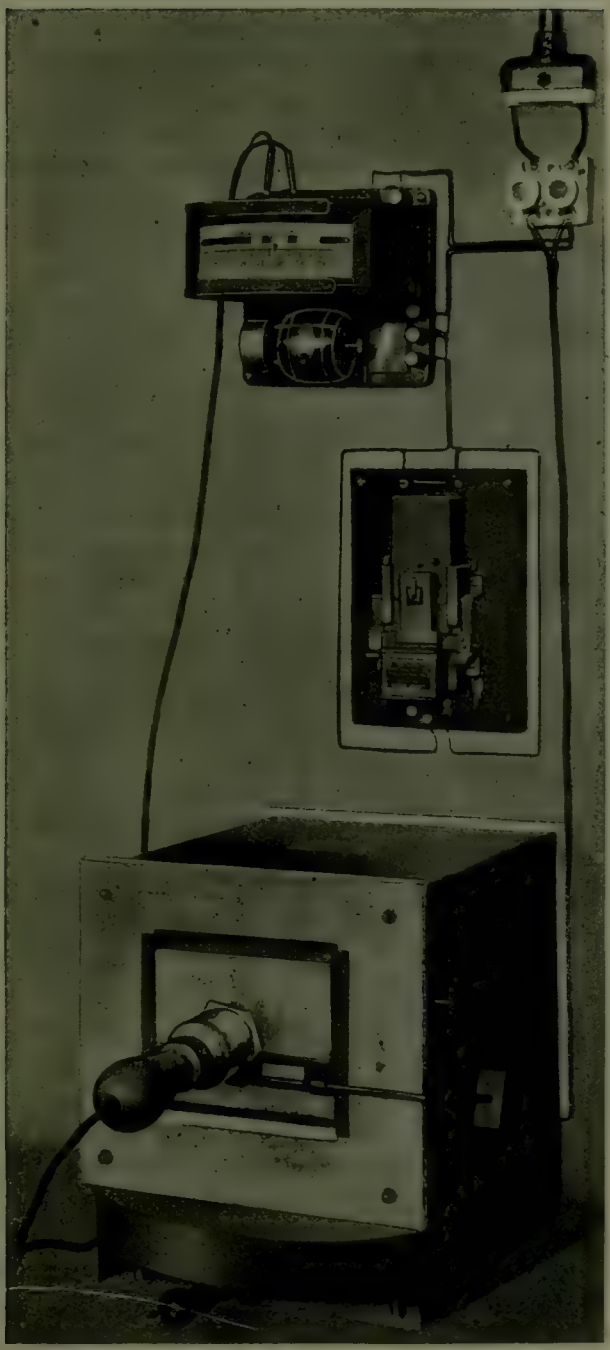


FIG. 106.—*Automatic temperature control pyrometer*

In industrial equipment usually the single-step method has been applied. In electric heating, the heating current is changed by opening or closing a switch, which will shunt a rheostat, change the series or parallel connections of the heater, or change the secondary side of a transformer. In gas or oil heating, the supply pipe is by-passed and the control valve is placed in this shunting line.

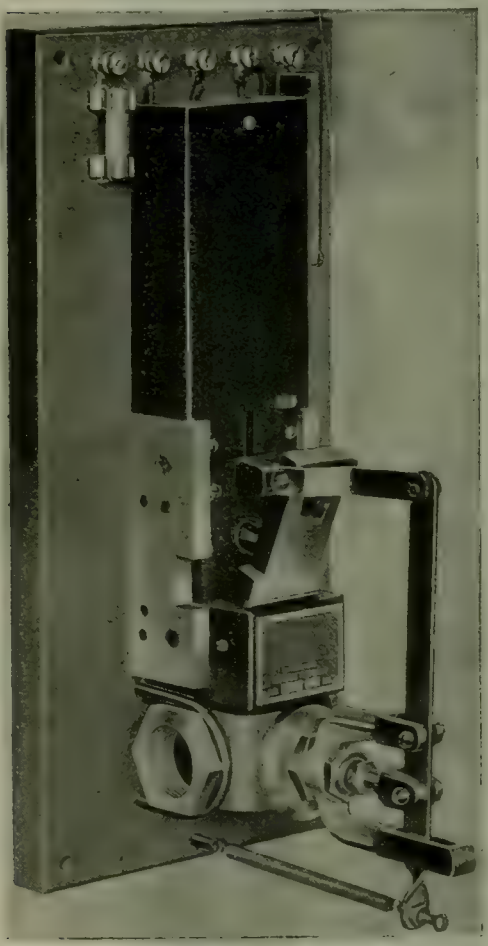


FIG. 107.—*Electromagnetically operated gas or oil valve*

Fig. 106 illustrates the Brown automatic temperature-control pyrometer, showing the galvanometer, motor for raising and lowering the depressor frame, and the solenoid switches. The galvanometer is the high-resistance type with 300 ohms for a base-metal couple and a scale range of 1100° C. The control is between limits 1 per cent of the scale range in extent; that is, it will control to about 10° for the above range. The range may be lowered considerably for closer regulation, say to 3° C, without serious disadvantage. By using a method for manual correction for resistance and low-resistance meter with a very open sale, the control may be easily perfected for less than a 1° variation. Obviously the cold junction of the thermocouple must be either buried deeply in the ground or placed in ice or a thermostated box. For temperatures below 425° C Brown substitutes a nitrogen-gas thermometer, which makes it possible to control to 0.2° and better, with a scale range of 10° or 15° C. Fig. 107 illustrates the form of the electromagnetically operated valve developed. The Bristol company has also adapted the instrument described for

signaling, so that it will control the furnace temperature automatically. This company has patented a valve somewhat different than the one illustrated, but designed to accomplish the same results.

An apparatus for automatically controlling the operation of electric furnaces for hardening steels, etc., has been recently developed.

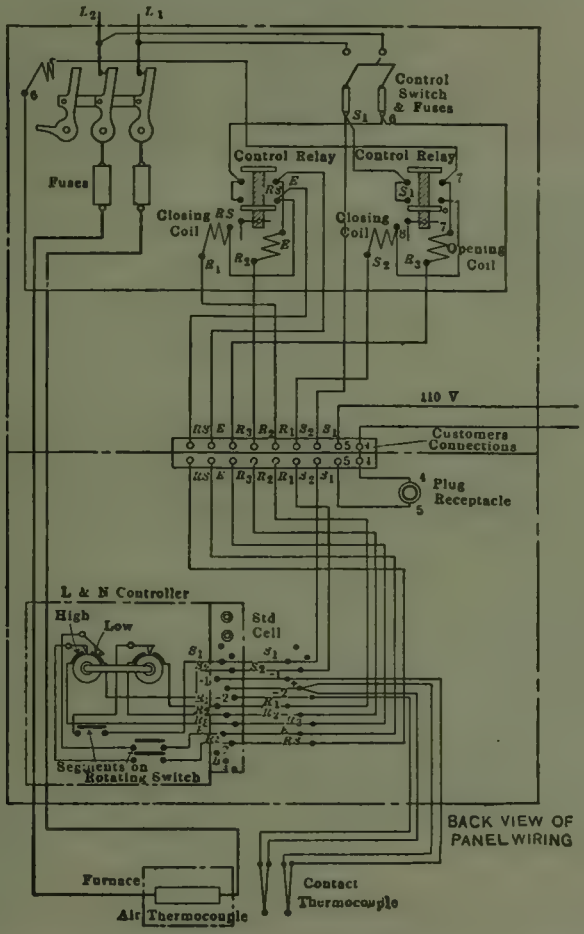


FIG. 108.—*Wiring diagram of automatic control panel for electric furnaces*

Fig. 108 is a wiring diagram of the control panel. This equipment automatically heats a hardening furnace up to a temperature, say 1000° C, somewhat above that desired, say 900° C, and holds it there until the piece being treated reaches 900°, when the furnace temperature is dropped to this value and held there. This unique operation is accomplished by placing one thermo-

couple, called the contact couple, in contact with the piece, and another, called the air couple, in the furnace near the wall. The air couple is kept hot until the contact couple reaches the proper temperature, when the air temperature is lowered to this value.

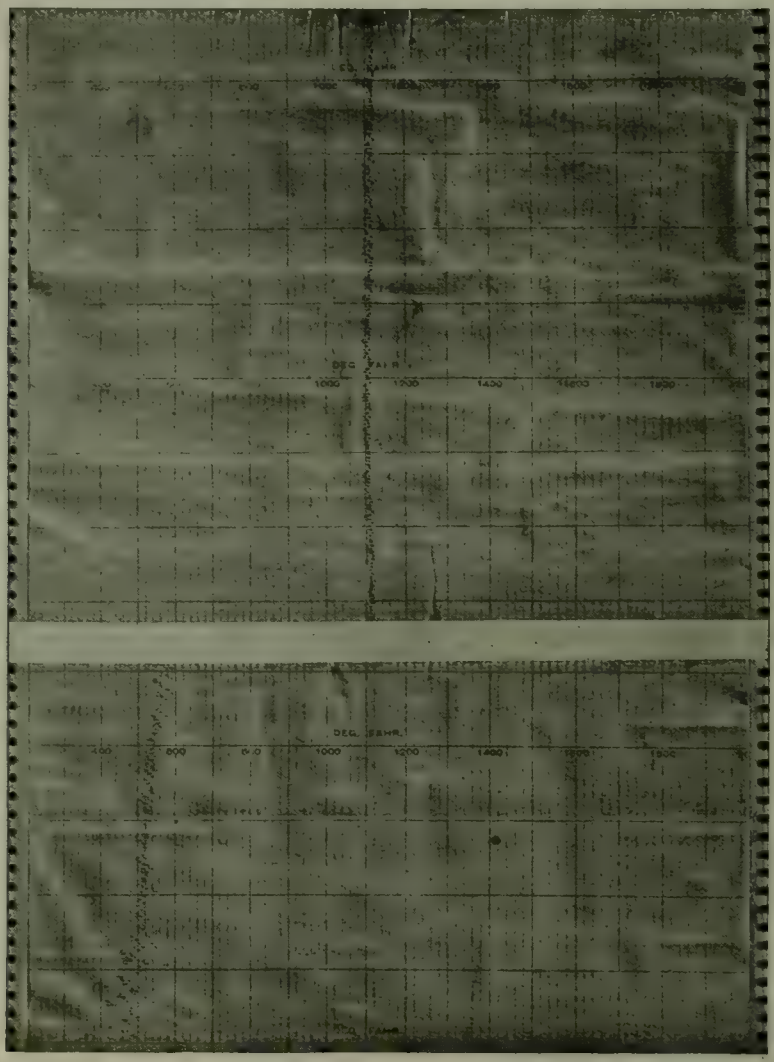


FIG. 109.—*Sections from record of 24-hour annealing heat*

The wiring diagram of Fig. 108 is clear upon close study of the connections and a discussion is scarcely necessary.[21] The con-

[21] In this diagram the following must be noted: (1) Control relays are closed when E is connected to 6 and S_1 is connected to 7. (2) Connecting S_1 and 7 closes the heating circuit by the magnetic switch on the left. (3) Contact thermocouple operates through contacts marked S_1, R_1, and R_2, and air-couple contacts S_2, R_3, RS and E. (4) S_1 and R_2 are connected and open a relay when contact couple reaches the desired temperature.

tacts operating the relays are opened and closed by the automatic adjustment of the potentiometer slide wire. By disconnecting either of these thermocouples and one of the control relays the instrument may be used simply to hold a furnace at a fixed temperature. Another application of the potentiometer type of automatic controller maintains the temperature of the box ovens used in japanning at the proper point. It is made to control at one temperature for a certain interval and then automatically changes to another temperature and maintains this.

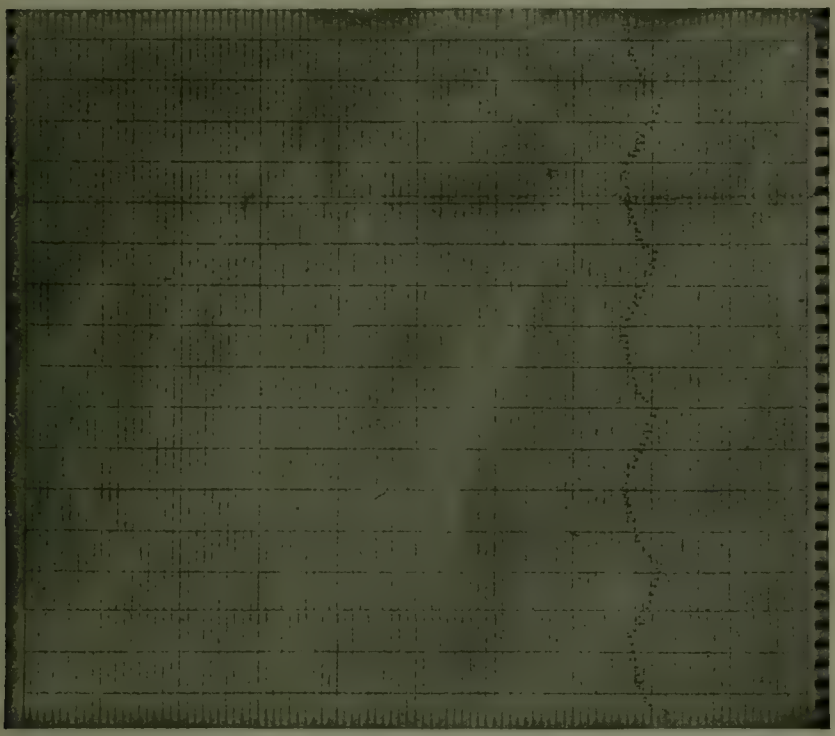


FIG. 110.—*Sections from record obtained of reversals in regenerative firing*

Still another operation can be performed with this instrument. One of the writers has constructed an apparatus which will heat or cool a furnace at a predetermined rate, either constant or a function of the temperature. This apparatus has been used in studying the annealing of glass, especially the fine annealing of optical glass. This type of control is obtained by placing one contact on the potentiometer slide wire and the other two contacts upon a disk which is made to turn slowly at a chosen rate. The disk is turned by the controlling recorder itself, so that the

whole apparatus is automatic. This principle of moving contacts is applicable to any of the controllers described, but at present such control has not been utilized industrially.

As a thermostat, the potentiometer-recorder controller can be made capable of operating upon 0.01° changes in the tempera-

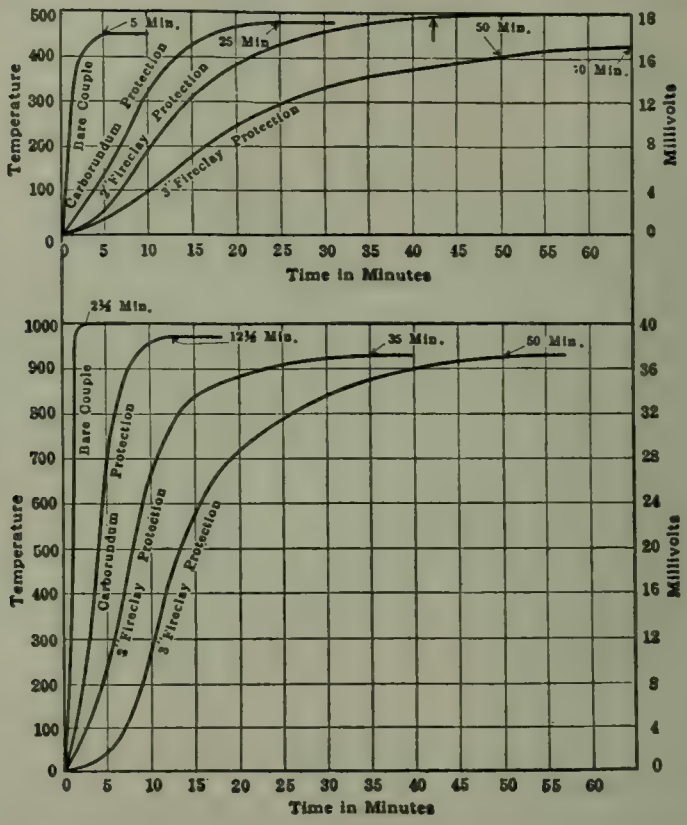


FIG. 111.—*Curves showing lag in pyrometer reading for thermocouples in various protection tubes*

ture of a resistance thermometer and upon less than 0.5° with base-metal thermocouples. Very rapid progress is properly expected in the future development of instruments for automatic temperature control at higher temperatures. The field is a comparatively new one and undoubtedly offers possibilities not fully appreciated at the present time.

2. INTERESTING EXAMPLES OF THE UTILITY OF RECORDERS IN FURNACE CONTROL

Fig. 109 is a reproduction of a chart from a six-point recorder showing the operation of a car annealer furnace 31 feet 4 inches long, 6 feet wide, and 44½ inches high from floor of car to top of rough arch. This furnace was fired from combustion chambers placed at the side and below the level of the car floor. The six points for which temperatures were recorded were in six different locations so chosen as to give the average condition of the entire heating chamber. The record shows how quickly after starting the operator was able to obtain a fair degree of temperature uniformity, and how at the last of the run the entire furnace was held for hours at a remarkably uniform temperature. This chart shows at a glance the condition desired, viz, uniformity.

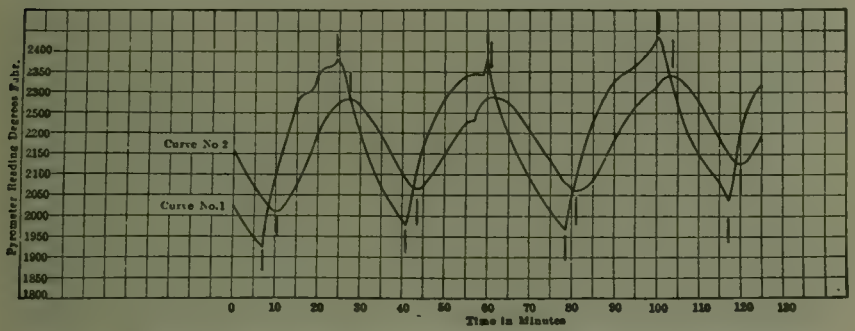


FIG. 112.—*Curves showing temperature variations of slag pocket of open-hearth furnace*

Curve No. 1, couple protected by 1⅜-inch carborundum tube. Curve No. 2, couple protected by 3-inch fireclay tube (Hutchins)

Fig. 110 is a simple example of the excellent record obtained of furnace temperatures during changes in regenerators. It illustrates the method of temporarily connecting one thermocouple to all the points of the recorder, thus securing a more complete record of the variations. If, for example, connections were made only to No. 1 point, the record obtained would be misleading. However, the precise shape of this curve has little meaning, for, as shown in Figs. 111 and 112, the record obtained depends considerably upon the characteristic lag of the thermocouple. [22]

Fig. 113 shows waste-heat temperatures taken when a furnace was changed from nonregenerative to regenerative firing. Properly marked with day and hour, this becomes a permanent record of the operation.

[22] Otis Hutchins, Pyrometer Protection Tubes, Trans. A. I. M. M. E., 153, p. 1811; Sept., 1919.

Fig. 114 is a record of experimental work done in a by-product coke oven. Five points of a six-point potentiometer recorder are in use, the sixth point being short-circuited in order to bring its record to zero. (At the beginning of the record, No. 6 left on

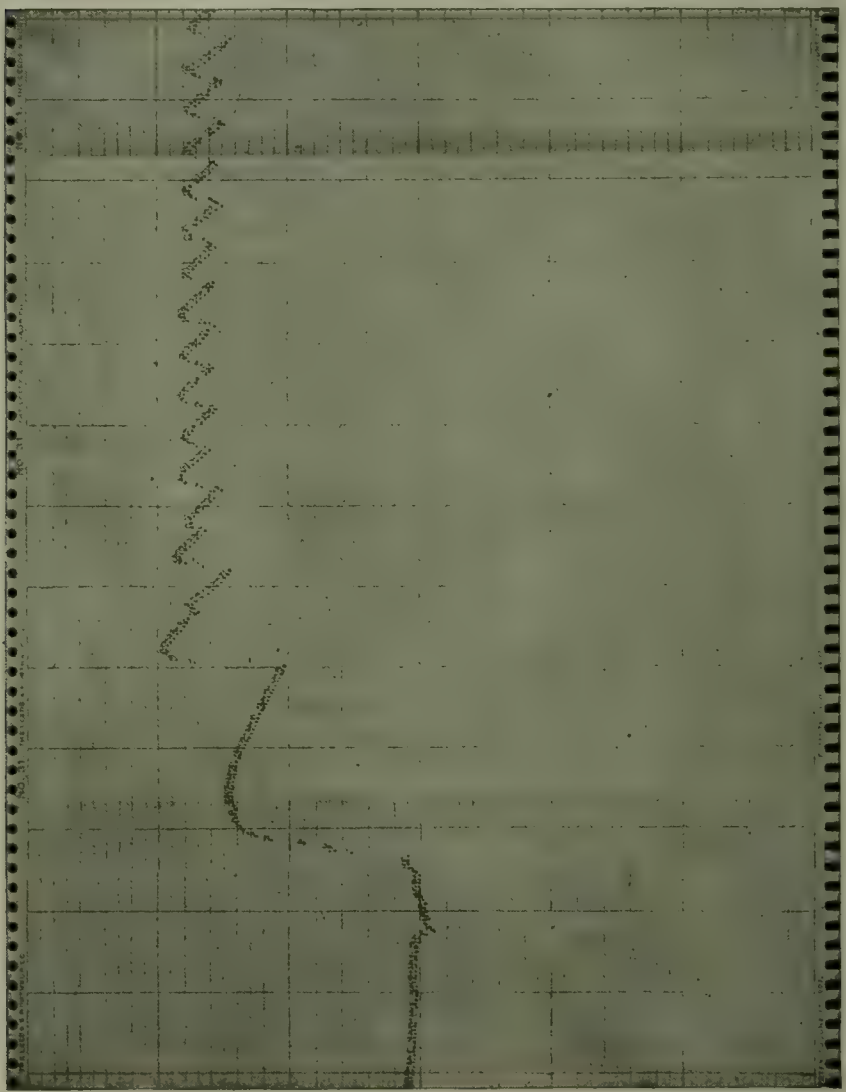


FIG. 113.—*Record of changes in method of firing*

open circuit became confused with No. 5.) This is a record of the last 13 hours of a "charge." No. 1 is the record of temperature of the vapors above the coal in the oven. Nos. 2, 3, and 4 are from thermocouples in the center of the coal charge inserted a

few hours before the temperature at this point rose above 100° C. At the end of the coking period couples 1, 2, 3, and 4 were withdrawn, and as the couples cooled the readings dropped to zero. No. 5 is the waste-heat record. This curve shows how incomplete

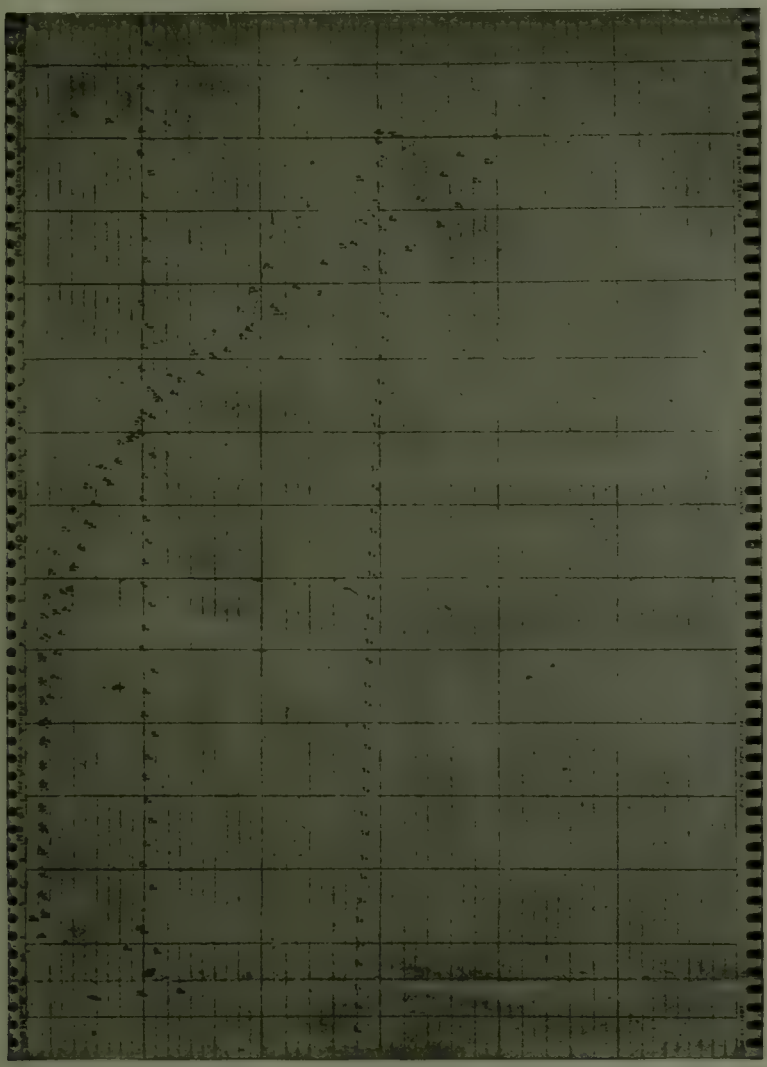


FIG. 114.—*Record of experiments in a by-product coke oven*

is a record obtained when the time interval between records is too long. The record of No. 1 is useful for a check on the operation of the recorder. Knowing that this record should show a gradual and uniform rise in temperature, any departure from a smooth

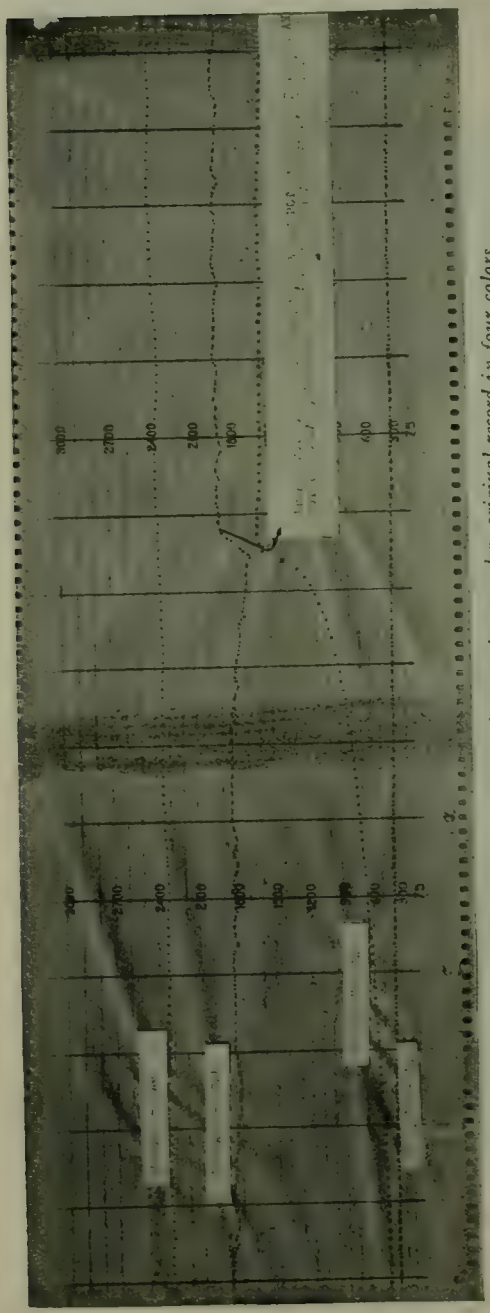


FIG. 115.—*Chart from 4-point galvanometer recorder, original record in four colors*

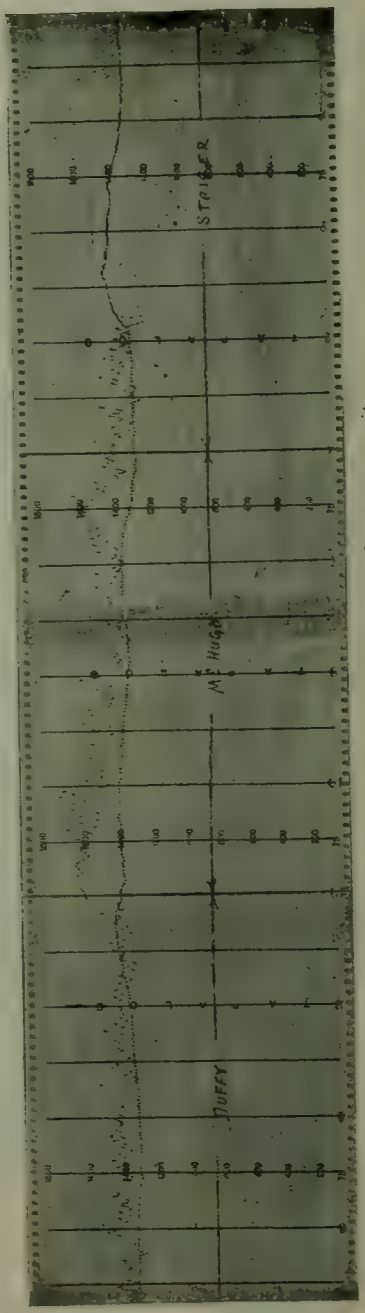


FIG. 116.—*Record for 24-hour run of water-gas unit*

curve is an indication of instrument trouble or tampering. For experimental work these curves are best replotted with the time coordinate shortened and a continuous smooth curve drawn through the points.

Fig. 115 is the chart from a 4-point galvanometer recorder with the thermocouples placed as indicated on the chart. The four records are made in colors and with dots differently placed, which makes them very legible. Operations are here clearly shown upon a permanent record.

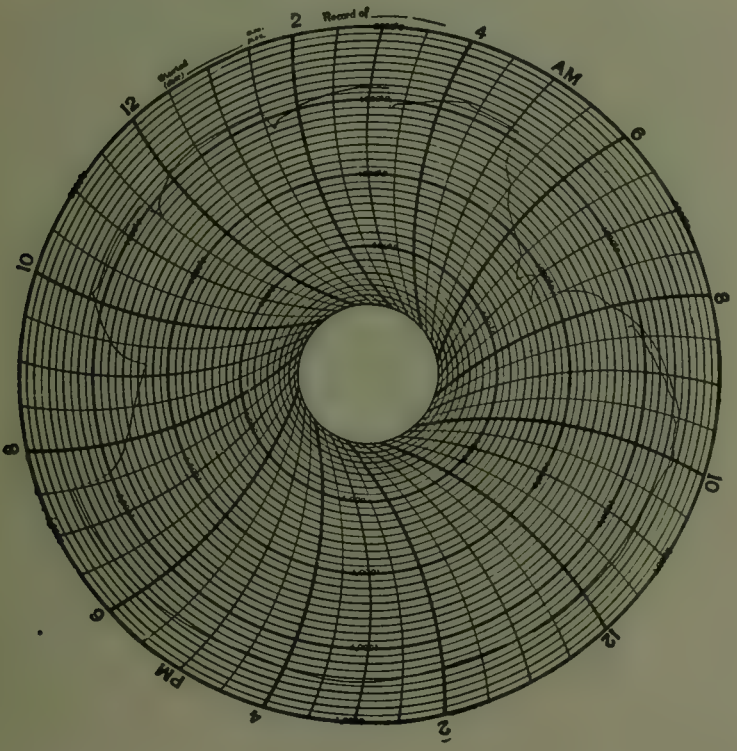


FIG. 117.—*Record of furnace used for case hardening during the day and annealing at night*

Fig. 116 is a record of temperatures in a water-gas unit. This record was selected to show how recorder charts may be used to indicate the comparative efficiency of operators. In this case the three shift operators, Messrs. Duffy, McHugh, and Staiger, were stimulated to better work when this record was framed and hung in the gas house. The lower curve should have been maintained within 25° of 1350° C and the upper curve between 1350° and 1550° C. Mr. Duffy very evidently secured the most satisfactory results.

Fig 117 is the record of the operation of a gas furnace used alternately for casehardening and for annealing. At 7.30 a. m. the furnace was charged and the temperature raised to approximately 1750° F at 9.30, where it was maintained with good control until 7.30 p. m. Then a new charge was put in, and the temperature was raised to 1575° F, this being repeated three times before 5 a. m , when the fire was cut off. This record was used by the operator for obtaining proper temperatures and serves

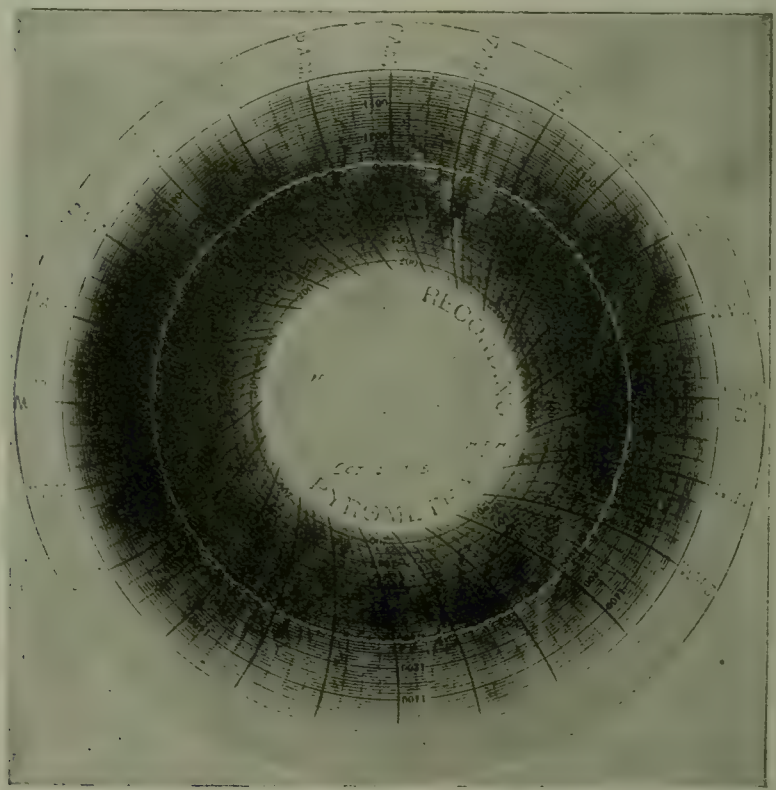


FIG. 118.—*Example of excellent control of annealing furnace (oil or gas)*

also as a permanent record of the number of heats. Knowing the processes at this plant, it is a record of what types of heat treatment were accomplished.

Fig 118 is an example of excellent control of a gas or oil furnace for annealing brass. Any laxity on the part of the operator would be very apparent in this record. However, such good control is exceptional. Fig. 119 is the record of stack gas temperature in a billet-heating furnace, and in this case the control was considered good.

Fig. 120 is another illustration of the clarity with which the relative efficiency of shift operators is shown. In this case the work of the day shift was superior to that of the night shift.

FIG. 119.—*Example of good control*

X. MELTING-POINT METHODS AT HIGH TEMPERATUERS [23]

1. INTRODUCTION

The determination of a melting or freezing point, whether for a physical or physical-chemical purpose, resolves itself into the measurement of a temperature under particular conditions and with appropriate technique. The accuracy and ease with which melting points at high temperatures may be determined is due to the comparatively recent developments in pyrometry, electric-furnace construction, and refractory materials.

The general classes of substances considered are metals, alloys, salts, and refractory materials. For the temperature measure-

[23] Dana and Foote, Chem. and Met. Eng., 22, p. 23 and p. 63; 1920. Also see Foote, Harrison, and Fairchild, same, 18, p. 343 and p. 403; 1918.

ments, thermoelectric, resistance, and optical pyrometers are employed, depending upon the temperature range and other controlling conditions.

The melting or freezing point of a pure crystalline substance is defined as the temperature at which the solid phase can remain in equilibrium with the liquid phase at normal atmospheric pressure. The effect of pressure is ordinarily insignificant; it is only when the pressure reaches many atmospheres that any change in the

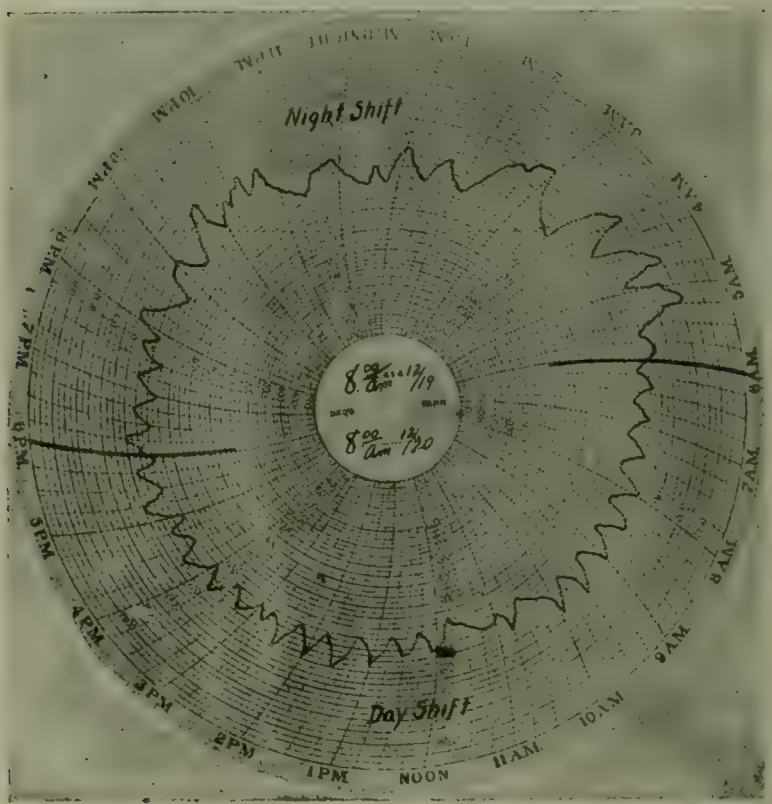


FIG. 120.—*Illustrating relative efficiency of day and night shift*

melting or freezing point can be readily detected. At the melting point there is a discontinuous change of a number of the physical properties of a material; e. g., thermal conductivity, electrical resistivity, specific volume, etc. In general, alloys, excepting eutectoids, mixtures of refractory oxides, and materials with impurities do not have a definite melting point, but become liquid gradually; that is, they melt over a temperature interval, and consequently are considered as having a "melting range."

2. USE OF THERMOELECTRIC PYROMETERS

(*a*) PURE MATERIALS—(1) *General Discussion.*—The experimental arrangement of this method consists of a crucible containing the substance in which a thermocouple either protected or unprotected is centrally inserted. The crucible is placed in a uniformly heated portion of an electric furnace. On account of cleanliness, clarity of the atmosphere, and preciseness of control, electric resistance furnaces are preferable to gas or fuel fired furnaces for accurate melting-point determination. When a melting or freezing point is to be observed, the temperature of the furnace is gradually raised or lowered, and the temperature as indicated by the thermocouple is noted at frequent, and preferably uniform, intervals of time. If all conditions were ideal, the temperature of the charge would remain constant during melting or freezing. Thus the temperature-time curve would be characterized by a straight line exactly parallel to the axis of time and by a discontinuous change of slope at either end of the line marking the beginning and ending of the period of melting or freezing. Actually, however, the change of state shown by the curve, even for pure materials, is not sudden, but more or less gradual. Only a part of the freezing curve will be flat, and the melting curve usually possesses greater obliquity than the freezing curve. For this reason freezing-point determinations are usually more reliable and are employed whenever possible, although the temperature thus obtained is frequently called the melting point, to which it is numerically equivalent.

The reasons for obliquity, and for its existence to a greater degree in the melting curves, have been discussed in detail by White.[24] On heating, the furnace walls are hotter than the crucible, and hence heat flows from the furnace to the crucible. When a layer of the charge near the wall of the crucible reaches the melting point, the heat absorbed by the charge goes to supply that necessary for the latent heat of fusion, and the temperature of the outside layer tends to remain fairly constant. The thermocouple measures the temperature of the solid metal in the center of the charge and when thus surrounded by an isothermal layer the rate at which the inside temperature increases is greatly diminished, resulting in a rounding off of the temperature-time curve. As the isothermal layer progresses inward the temperature of the couple slowly rises until the material immediately surrounding it

[24] White, Melting-Point Determinations, Am. J. Sci., 28, p. 453; 1909.

begins to melt, when a condition of stationary temperature is maintained for a short time during which the thermocouple reading gives the true melting point. While the center of the charge is melting, however, the temperature of the outer layer of material in the crucible rises very rapidly, and a large temperature gradient is established between the outside and center. The resulting rapid flow of heat to the center of the crucible accelerates the melting and, if the layer of remaining solid metal is unequally distributed about the couple, tends to increase the temperature readings and cause another rounding off of the temperature-time curve. The apparent degree of obliquity found near the beginning and end of the melting period depends considerably on the sensitivity of the temperature measurements and scale of plotting. Thus, highly accurate data obtained with a precision potentiometer may be plotted on so large a temperature scale that the curve may appear quite oblique, while the same data replotted on a less extended scale appear fairly flat. Aside from this, however, the degree of obliquity varies according to the factors controlling the differences in temperature within the charge, such as dimensions and thermal properties, rate of heat supply, etc. If the solid metal could be stirred during a melting-point determination the temperature gradients could be diminished, but even with powdered materials where stirring is possible it is not convenient and does not appear to be very satisfactory. The larger the diameter of the charge, the faster the rate of heating, the smaller the diffusivity, and the lesser the latent heat of fusion, the greater will be the tendency to obliquity. The obliquity may be decreased by slow heating and by using a charge of fairly small diameter. The protection tubes of the thermocouple should be small, of thin wall, and should be deeply immersed in the charge. With metals the obliquity is not so great as with salts, on account of their higher heat diffusivity and latent heat of fusion; in fact, for most cases the obliquity is of negligible importance with pure metals. But with salts and certain refractory materials, unless special care is taken, the obliquity may be so great as to almost completely obscure the true melting phenomenon.

On cooling, the crucible is hotter than the furnace walls, and heat flows from the crucible to the furnace. The outer layer of the charge solidifies first and during freezing acts as an isothermal layer, thus decreasing the temperature differences between the center and outside of the charge. The rate at which the temperature

of the couple falls decreases, causing a rounding off of the first part of the temperature-time curve. While the center of the charge is freezing, the outside is completely frozen and rapidly falls in temperature, increasing the temperature gradient and the outward flow of heat, and hence the latter part of the freezing is accelerated.

The first part of the approximately flat portion of the curve for freezing corresponds to the true freezing point, and the latter part of the approximately flat portion of the curve for melting corresponds to the true melting point. The same precautions of high narrow charges, narrow protecting tubes, sufficient depth of immersion, and small thermocouple wires apply to both cases.

In general, the freezing curves are more sharply defined than the melting curves. A liquid cooling and subject to convection currents is likely to possess a more uniform temperature distribution than a solid on heating. Furthermore, the freezing point is chosen at a time immediately following a period of small temperature gradients, while the melting point must be chosen near the end of the period of melting when there is possibility of temperature non-uniformity. A further factor of importance is the constancy of heat supply. On cooling with a furnace well insulated, the freeze may extend over a considerable time with all power supply shut off. The furnace thus acquires a uniform rate of cooling. In heating, however, the effect of slight variations in the power supply may alter the true character of the melting curve. Fig. 121 shows a freezing and a melting curve in which considerable obliquity is present. The true temperature for freezing is obtained by extrapolating the straight portion of the freezing curve and noting at what temperature *A* the continued straight line deviates from the observed curve. The melting point *B* is similarly obtained from the melting curve. The first part of the freezing curve and the last part of the melting curve determine these two temperatures as stated above.

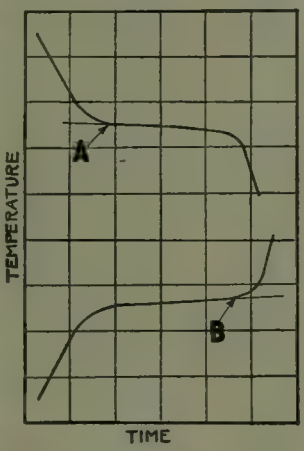


FIG. 121.—*Oblique freezing and melting curves*

It is impossible to obtain satisfactory freezing curves with certain materials, such as silicates, on account of extremely slow crystallization, and the melting curves, even though frequently poorly defined, must be employed. The phenomenon of surfusion

or supercooling is well illustrated by water which may be cooled to $-30°$ C without freezing. Surfusion is due to conditions which do not favor the attainment of equilibrium between the solid and liquid phases at the true freezing point. The conditions tending to produce equilibrium and the elimination of surfusion are slow cooling, stirring of the liquid and the presence of nuclei which serve as centers of crystallization. When a supercooled liquid freezes, it crystallizes very rapidly and the temperature rises to the true freezing point provided the supercooling has not proceeded too far. The subsequent freezing takes place in a normal manner. Surfusion in most metals seldom exceeds 0.1 or 0.2° C, but in the case of antimony it is very marked and may amount to 30° C, depending upon the rate of cooling, as shown by Fig. 122. Supercooling also occurs in alloys, especially in eutectics. While the contrary phenomenon of superheating is negligible with pure

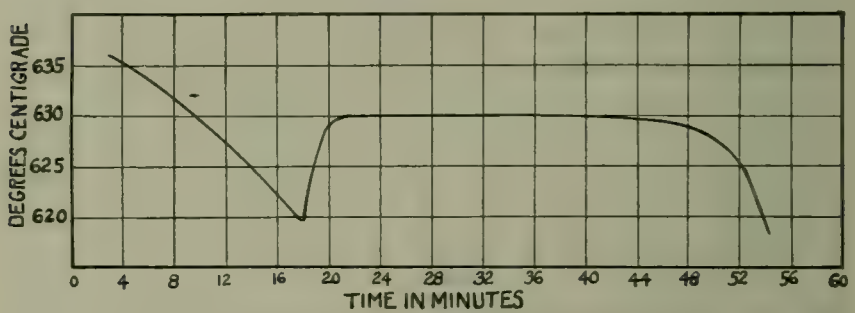


FIG. 122.—*Freezing curve of antimony, showing undercooling*

metals, it may become of importance with alloys, particularly in allotropic transformation and with salts and silicates.

(2) *Precision Work with Pure Metals.*—The primary method for the standardization of thermocouples consists in determining the emf developed by the couple at the freezing points of several very pure metals. The metals so employed are those the freezing points of which have been accepted as fixed points on the high temperature scale; e. g., tin, cadmium, zinc, antimony, aluminum, silver, gold, copper, etc. For this work and for the reverse problem of accurately determining the freezing points of metals and alloys the greatest precautions against contamination of the couple and the metal are requisite. Precision work at high temperatures requires the use of homogeneous platinum, 90 per cent platinum-10 per cent rhodium couples. The diameter of the wire usually employed is 0.5 or 0.6 mm, but for certain special purposes wires of 0.1 mm are useful.

(3) *Crucibles and Protecting Tubes.*—Of all crucible materials Acheson graphite has the greatest utility. It is very pure, can be machined into any desired shape, and is not attacked by most of the common metals. At high temperatures the gases formed from its oxidation provide the reducing atmosphere necessary for the protection of the metal. The following table illustrates sizes of graphite crucibles, which have been used in various investigations. For resistance thermometry the largest size shown is necessary, but for general work the intermediate size is recommended. In this case a smaller protecting tube is desirable—for example, one with an outside diameter of 5 mm.

Some metals, such as nickel and iron, combine with graphite, and certain other metals also should not be heated under the reducing conditions present with graphite. Crucibles of magnesia alumina, or mixtures of the two, are suitable for iron and nickel. Porcelain crucibles may be used for many of the metals, but there is always danger of these cracking when the metal melts or freezes.

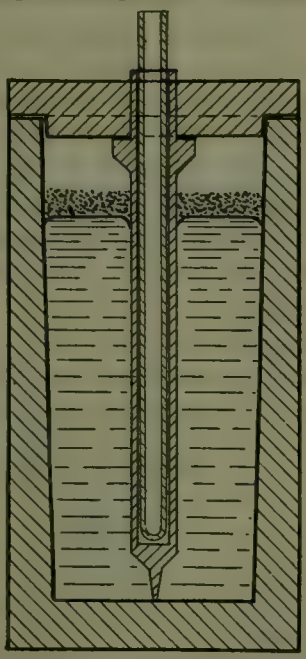


FIG. 123.—*Graphite sheath for protection of porcelain tube in aluminum*

TABLE 23.—Size of Crucibles

Laboratory	Crucible dimensions, all in centimeters				Charge dimensions		Protection tube		Distance from bottom of charge to protection tube
	Inside		Outside		Diameter	Height	Diameter		
	Diameter	Height	Diameter	Height			Outside	Inside	
Bureau of Standards	7.0	15	8.5	16	7.0	11	1.0	0.8	1.0
Geophysical laboratory	2.7	8	3.7	10	2.7	5	.8	.5	.5
Bureau of Standards	1.2	3	2	3.5	1.2	2	.15	.1	.3

Up to 500° C protection tubes and insulating tubes for the couple, made of pyrex glass, are useful. From 500 to 1100° C lower grade porcelain, glazed on the outside only, or fused quartz,

are satisfactory. Above 1100° C tubes having a composition approximating that of sillimanite, Al_2O_3, SiO_2, are recommended.[25] Porcelain tubes, or crucibles, or any material containing silica, can not be used in contact with aluminum, as the silica is readily attacked. Aluminum may be melted in a graphite crucible, and the porcelain protecting tube may be itself protected by a very thin sheath of graphite. Fig. 123 illustrates one convenient manner in which the sheath may be mounted in the crucible. The sheath is held down in the metal by the weight of the cover and is allowed to remain in the crucible after the aluminum is frozen. The thermocouple-protecting tube fits tightly inside the sheath. At present there is no satisfactory protecting tube for nickel. This material and its alloys may be best studied by optical methods.

(4) *Table of Melting Points.*—The following table lists the more common metals, the melting points of which can be used for the standardization of thermocouples by the crucible method:

TABLE 24.—Melting Points of Metals from Tin to Nickel

Metal	Melting point	Crucible material	Atmosphere
	°C.		
Tin	231.9	Graphite, porcelain, or pyrex glass	Reducing or neutral
Cadmium	320.9	do	Do.
Lead	327.3	Graphite	Do.
Zinc	419.4	do	Do.
Antimony	630.0	do	Reducing, CO
Aluminum	658.7	do	Do.
Silver	960.5	do	Do.
Gold	1063.0	do	Reducing, neutral, or air
Copper	1083.0	do	Reducing, CO
Nickel	1452.0	MgO, Al_2O_3 or mixtures of the two	H_2, N_2, or vacuum

The surface of the metal, except nickel, should be covered with a layer of powdered or flaked graphite, and the crucible should be provided with a cover having a hole through which the thermocouple tube is inserted. Under no circumstances should graphite be exposed to an air current above 500° C, as it readily oxidizes and crumbles away. The furnace should be tightly closed at the bottom and no larger openings exposed at the top than absolutely necessary. When fairly small graphite crucibles are employed, it is frequently desirable to use a larger outside crucible of porcelain and fill the space between the two crucibles with

[25] Such porcelain is known as Usalite or Impervite.

graphite powder, as shown in Fig. 124. When larger crucibles are used, the graphite powder may be poured directly into the furnace tube to a depth sufficient to entirely cover the crucible.

(5) *Construction of Furnace.*—Fig. 124 shows the construction of a furnace using crucibles of the intermediate size given in Table 23 for operation up to 1100° C. The graphite crucible *H*, containing the metal *G* and the pyrometer tube *A*, rests in a porcelain crucible *F* and is completely covered by powdered graphite *E*. The porcelain crucible is supported by the alundum tube *I*. The heater tube is of alundum, R. A. 98; length, 25 cm; inside diameter, 5 cm; and wall thickness, 3 mm. This is preferably corrugated on the outside and is wound with 60 to 80 turns of No. 14 "chromel A" wire, *C*, to the ends of which are welded the lead-in terminals *K* of No. 10 chromel. The alundum tubes *B* and *I* rest on a good nonconducting fire brick about 5 by 8 by 8 cm. The shell *M*, 32 cm high, is of sheet iron or monel metal painted with aluminum paint. The ends of the shell are closed by impregnated asbestos boards *N*, 1 cm thick and 17 cm in diameter. The annular space *O* is packed with silocel powder. The lead-in wires are insulated from the metal shell by asbestos board or steatite bushings. The furnace in series with a rheostat for control may be operated on 110 volts and consumes about 1 kilowatt at 1100° C.

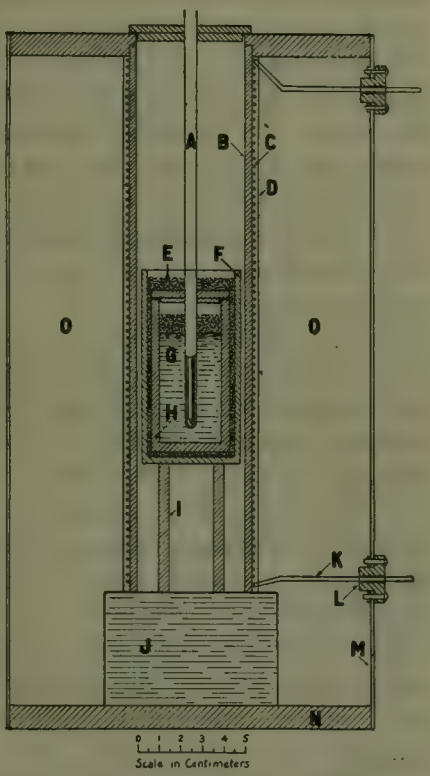


FIG. 124.—*Melting point furnace for metals and alloys*

Larger furnaces are required for the large crucibles described in Table 23. Such a furnace may be constructed from the following stock material. Alundum tube, R. A. 98; length, 45 cm; inside diameter, 8 cm; wall thickness, 6 mm. Chromel A winding No. 5 wire; 60 turns separated by and covered over with

alundum cement. Diameter of shell, 36 cm; height of shell, 60 cm; silocel powder insulation, general construction similar to Fig. 124. Power required for 1100° C about 55 amperes at 60 volts. Using the large crucibles a freeze of copper with all power off extends over a period of about 20 minutes.

(6) *General Precautions.*—In order to melt for the first time a metal which is in small pieces, the graphite crucible, filled with the material, fitted with a cover having a hole in which the thermocouple fits tightly and surrounded by powdered graphite, is slowly heated. If convenient, CO or N_2 may be passed into the furnace, but CO_2 should not be used. The surface of the metal should not be covered with graphite at first, as it may filter into the interior and become imprisoned there. When the metal has been melted, however, the graphite layer on the surface is quite desirable. With the exception of antimony, which should be stirred just as the freezing point is reached to reduce supercooling, it is not advisable to stir the molten metals, as pockets of graphite are thereby formed.

In melting large crucibles of copper care must be taken that the furnace is not burned out before the copper is melted. It is frequently desirable to use a thermocouple connected to the heating element attached to a temperature control apparatus and arranged to prevent the winding being heated above 1200° C.

If there is doubt concerning the proper depth of immersion, several determinations should be made at different depths until a position is found where a slight variation does not affect the temperature measurement.

The pyrometer tube may be allowed to remain in the metal for a few degrees below the freezing point without breaking, and if it can not be removed then, the metal should be immediately remelted.

A uniform rate of cooling of about 2° C per minute from 10° C above the melting point is satisfactory for a freezing-point determination. The values obtained from melting and freezing curves should agree to from 0.1 to 0.01° for pure metals, and the curves should have a flat portion extending over at least 10 minutes. If the curve is not fairly flat the metal is likely impure, and hence possesses a melting range instead of a true melting point. Fig, 125 illustrates an observed freezing and melting curve for zinc. The freezing curve is constant to 0.03° C for a period of 15 minutes and the melting curve for 10 minutes. The obliquity in the latter part of the melting curve amounts to 0.1° C in six minutes.

(*b*) METALS WITH IMPURITIES, AND ALLOYS.—An impurity in a metal usually lowers the melting point and produces obliquity in the melting and freezing curves, the magnitude of both of these effects increasing, within limits, as the amount of impurity is increased. Assuming that the amount of impurity is small and that an ideal solution with the metal in the liquid state is formed, the lowering of the freezing point Δt may be expressed approximately by the Raoult-Van't Hoff equation:

$$\Delta t = \frac{R\vartheta^2}{L}\frac{N_1}{N},$$

in which $R = 1.99$, ϑ = absolute temperature of the freezing point of the pure metal, L = molar latent heat of fusion of the pure metal, N_1 = number of mols of impurity, and N = number of mols of pure metal. In Fig. 126, *A* represents the freezing curve of a pure substance and *B* the curve obtained when an impurity is present. The lowering of the freezing point is 4° C, an amount readily caused by a few tenths of a per cent of an impurity at about 800° C. In general, the above equation for freezing-point lowering holds only for an impurity which forms the simplest type of eutectic alloy with the pure metal. Evidently it can not be applicable to those impurities which are wholly or partially soluble in the metal in the solid state, that is, which form solid solutions, for in some of these cases the melting point is raised by the addition of the impurity, as, for example, nickel added to copper.

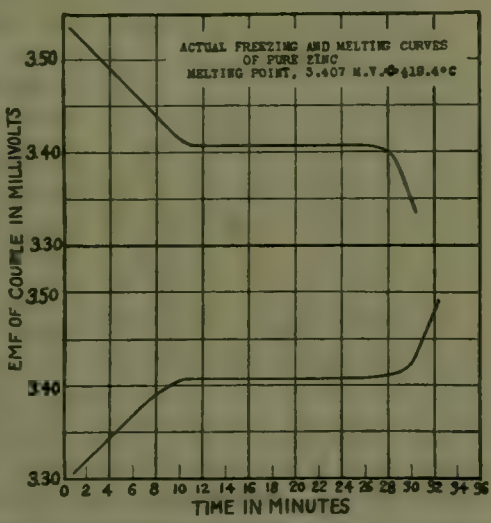


FIG. 125.—*Freezing and melting curves of pure zinc*

For most of the common metals the oxide is insoluble in the pure metal, but with copper this is not the case, and special care must be taken, preferably by the maintenance of a strongly reducing atmosphere, to avoid the formation of cuprous oxide Cu_2O. This oxide forms a eutectic with copper saturating it at 3.45 per cent of Cu_2O or 0.395 per cent of oxygen by weight, and freezing

at 1063° C. As an example of the application of the above equation, the freezing point lowering of copper at the eutectic, considering the Cu_2O as the dissolved impurity, will be calculated.

$$\Delta t = \frac{R\vartheta^2}{L}\frac{N_1}{N} = \frac{2(1083+273)^2\,(2.19)}{(63.6)(43.3)(143)} = 20.4^\circ\,\mathrm{C}$$

The lowering, found experimentally, is 20.0° C.

If the copper contains only a small amount of the oxide, the first solidification begins slightly below the melting point of pure copper and crystals of copper separate from the solution. The concentration of the dissolved impurity Cu_2O, accordingly, increases so that the freezing point of the remaining part of the solution is still lower. The pure copper will continue to separate from the liquid at a gradually decreasing temperature until the solution becomes saturated with Cu_2O. This results in rounding or obliquity of the cooling curve. When the remaining solution is saturated, both the Cu and the Cu_2O solidify together at a constant temperature as a eutectic mixture. At this point a halt occurs in the cooling curve, the magnitude of which depends upon the amount of the eutectic present. The effect of a small amount of Cu_2O will escape notice in an ordinary melting-point determination; but, if considerable oxide is present, the freezing curve shows the two retardations—one below 1083° and the other at 1063° C. Observation continued through the latter point may serve as a rough criterion of the purity of copper suspected of contamination by its oxide. The obliquity of the curve also may be an indication of impurity. If a copper wire is slowly heated in air the melting usually begins at the eutectic point, 1063° C, instead of at 1083° C.

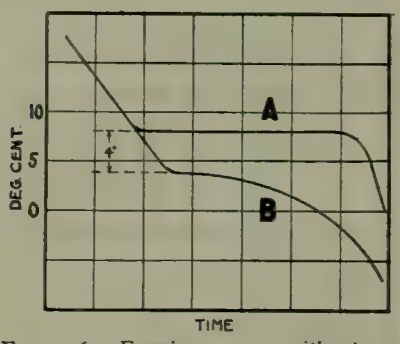


FIG. 126.—*Freezing curves without and with impurity*

Oxygen dissolves in silver and may lower the melting point as much as 5° C if the silver is heated in air.

An alloy may be considered as a metal containing large proportions of one or more impurities. Let us consider the simplest case of an alloy of two metals, *A* and *B*, which are mutually soluble when liquid and insoluble when solid. If *A* first begins to separate

from the solution, the remaining liquid becomes richer in *B*, and, as shown above, this part of the freezing curve gradually changes in slope, as illustrated by Fig. 127, curve *I*, *L*. As the temperature is lowered the solution finally becomes saturated with *B*, and *A* and *B* crystallize together as a eutectic, shown in Fig. 127, by the lower flat portion of the cooling curve. The alloy accordingly has a melting or freezing range extending from the temperature at which separation of *A* first begins to the temperature at which the eutectic mixture solidifies. If the original composition of the alloy is the eutectic mixture, there exists a true melting point as shown in Fig. 127, curve *II*, and not a melting range.

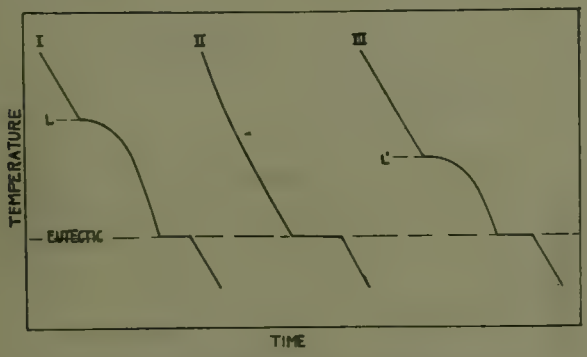


FIG. 127.—*Freezing curves of simple eutectic alloys*

The general form of the temperature-composition diagram of the simple binary alloy described above is illustrated by Fig. 128. The curves *I*, *II*, and *III* of Fig. 127 show the form of the cooling curves obtained with the alloys of compositions *I*, *II*, and *III* of Fig. 128.

Often a binary alloy exhibits two halts in the melting and freezing curves—one oblique and one flat or both oblique. Furthermore, other complications may arise, such as the formation of solid solutions, compounds, and allotropic transformations accompanied by heat changes which require more sensitive methods of detection than by a simple cooling curve. In general, the larger the melting range in a binary alloy the more difficult it becomes to locate the lower transformation point because of the small amount of crystallization per degree drop in temperature and the resulting small heat effect. The same general details for melting point of pure metals apply to alloys. The eutectic alloy of silver and copper, 71.9 per cent Ag, 28.1 per cent Cu, by weight, melts at

779° C and is useful as a standardization temperature for thermocouples.

For alloys having a large melting range accurate control of the rate of heating and cooling is essential. Automatic control of the rate may be obtained by a salt-water rheostat as applied by Burgess and Crowe,[26] or an automatic magnetic control system may be installed. Scott and Freeman [27] describe a modified Rosenhain furnace especially useful for critical point work. This consists of a vertical tube furnace, heated at the top and cooled at the bottom, through which the material under investigation is automatically moved at a definite rate.

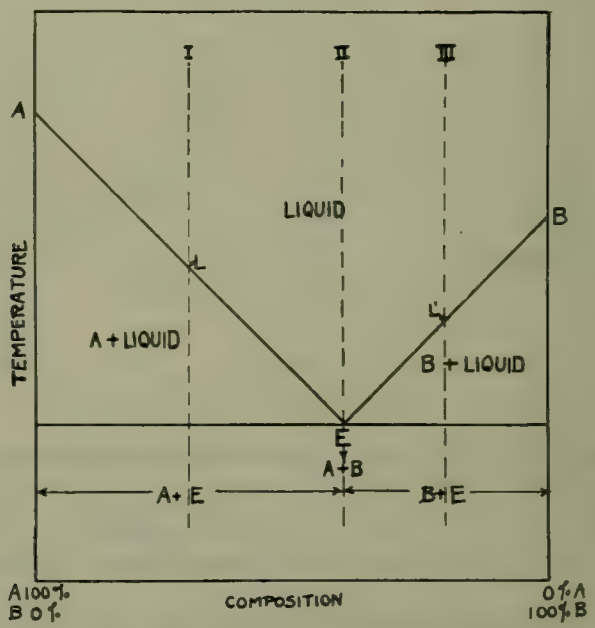


FIG. 128.—*Temperature-composition diagram of simple eutectic alloy*

If the ordinary temperature-time curve does not give evidence of a change of state at the beginning or end of a melting range or at a transformation point, the more sensitive means of detection, such as the inverse-rate method and the differential method, may be employed.

The inverse-rate method consists in determining the time required for the specimen to fall a definite temperature interval—for example, 2° C—and in plotting this time against the tempera-

[26] B. S Sci. Papers No. 213.
[27] Bull. Am. Inst. Min. and Met. Eng.; Aug., 1919.

ture. In effect, it amounts to differentiating the temperature-time curve in respect to temperature. The experimental arrangements are the same as for the ordinary-time method, except that the time must be measured more accurately. Merica [28] has described a simple and effectual device consisting of two stop watches, one of which is stopped and one started at the end of

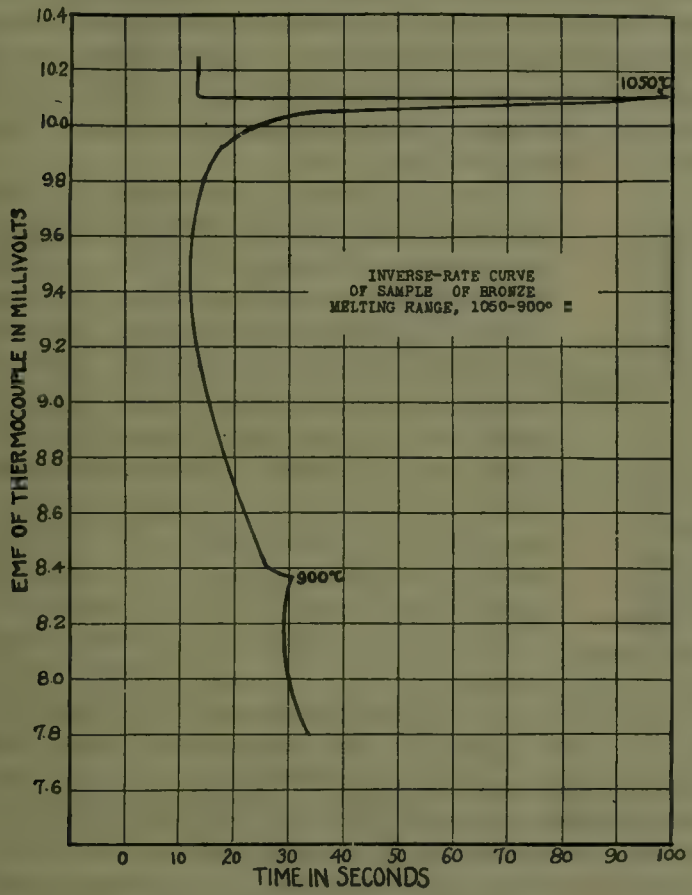


FIG. 129.—*Inverse-rate curve of a bronze*

each time interval. Fig. 129 shows an inverse-rate curve through the melting range of a bronze. The lower transformation at 900° C could not be detected by the ordinary temperature-time method.

The differential method is still more sensitive to small thermal changes. This consists in measuring the temperature dif-

[28] Bull. Am. Inst. Min. and Met. Eng.; July, 1919.

ferences between the material in which the transformation takes place, and a neutral body having no transformation points in the same temperature interval, such as platinum or nickel, and plotting this difference against the temperature of the material. Two thermocouples are employed as illustrated by Fig. 130, one a differential couple and the other an ordinary couple which measures the temperature of the sample under investigation. By use of a sensitive galvanometer with the differential couple extremely minute thermal transformations may be easily detected. Since the material under test and the neutral body are placed in close proximity in the furnace, slight variations in the rate of heating produce no noticeable effect.

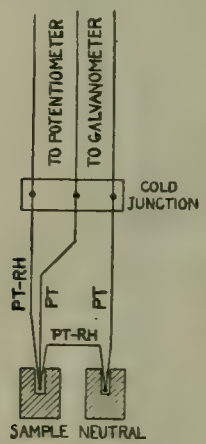


FIG. 130.—*Diagrammati representation of differential method*

Another method of interpreting the data is by means of the derived differential curve, first suggested by Rosenhain.[29] This amounts to differentiating the differential curve in respect to the temperature and plotting the value so obtained against the temperature. The derived differential is accordingly related to the differential curve in the same manner that the inverse rate is related to the temperature-time curve. Thus, if ϑ is the temperature of the material being investigated and ϑ' the temperature of the neutral body, the derived differential is the graph $d(\vartheta-\vartheta')/d\vartheta$ versus ϑ.

The method consists in observing successive values of $(\vartheta-\vartheta')$ for equal increments of ϑ, say $2°$ C, and plotting successive differences in these values of $(\vartheta-\vartheta')$ against ϑ. The experimental arrangement is the same as that required for the differential method. Fig. 131 shows both differential and derived differential curves for the same sample of pure iron at the transformation points A_2 and A_3.

(*c*) INORGANIC SALTS AND SILICATES.—On account of the low heat diffusivity and latent heat of fusion of salts and silicates, considerable difficulty is encountered because of the obliquity of the melting or freezing curves. Consequently, it is necessary to use tall, narrow charges and slow rates of heating. Few salts can be heated in a reducing atmosphere and hence graphite crucibles are prohibited. Porcelain and fire clay are attacked by most salts, so that metal crucibles must be employed. Of these, plati-

[29] Burgess, B. S. Sci Papers No. 99.

num, platinum alloys, and nickel have the widest utility. The thermocouple protecting tube of porcelain must be itself protected by a thin-metal sheath. In many cases salts do not attack platinum perceptibly, so that the thermocouple may be immersed directly in the molten salt. When this is done the crucible must be free from volatile metals, such as iridium, especially for work at high temperatures; otherwise the couple will become contaminated. If no protecting tube is employed, very small crucibles may be used, from 1 to 2 cm in diameter and 2 to 4 cm high. If

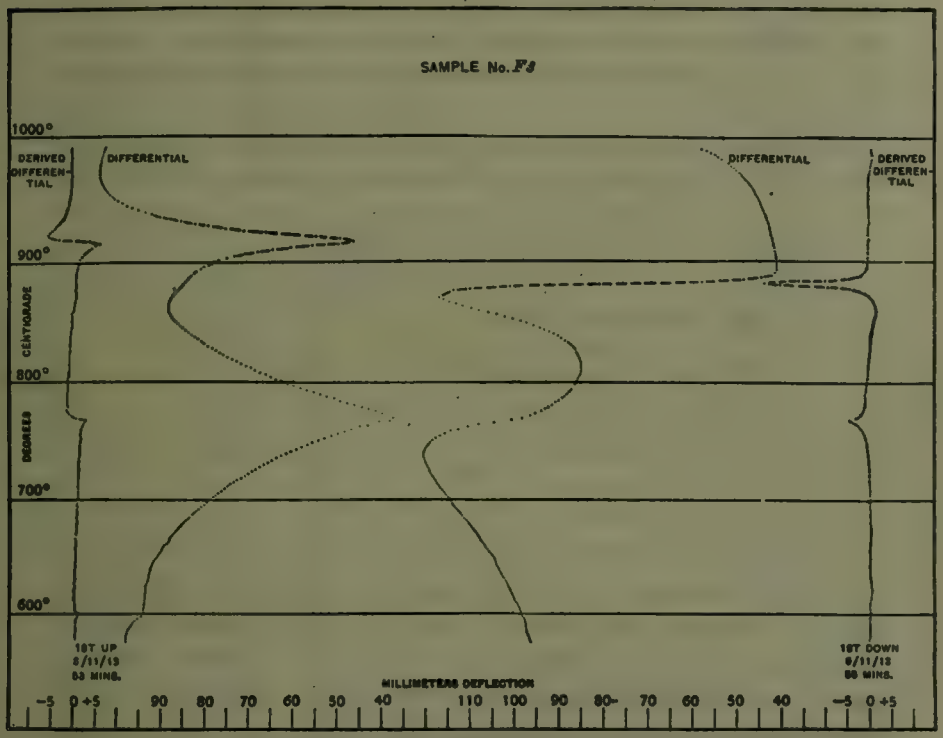


FIG. 131.—*Comparison of differential and derived differential methods*

the salt is quite volatile and the vapors distill into the furnace an additional protection tube for the furnace heater is necessary. The melting or freezing points of the nitrates, chlorides, and sulphates of the alkali and alkali earth metals may be determined in the above-described manner.

The melting points of silicates and other compounds of metallic oxides are usually comparatively high and their great viscosity when liquid, slow melting, slow crystallization, and under-cooling introduce many difficulties in the melting-point determination.

Many crystalline silicates which have a definite melting point do not show a definite freezing point on account of the high viscosity of the liquid, thus preventing the formation of crystal nuclei. The liquid supercools and the viscosity may so increase that a glass is formed; thus borax may be melted but once, and on cooling a clear glass results, which with reheating shows no marked heat transformation at the melting point. Other materials acting in this manner to a lesser degree are diopside, $MgSiO_3.CaSiO_3$, melting at 1391° C, and lithium metasilicate, Li_2SiO_3, melting at 1202° C. Consequently, in silicate melting-point determinations the melting curves are almost exclusively considered. In Fig. 132, *A* represents about as good a curve as is usually obtained with a silicate.

In another class of silicates the melting requires a large time interval, probably on account of the high viscosity of the liquid,

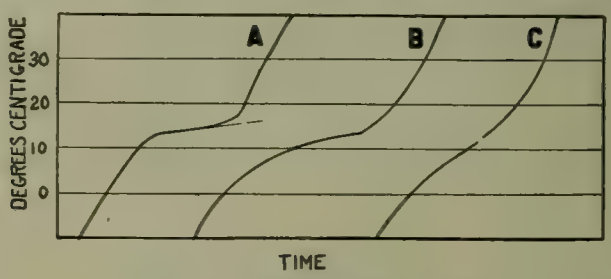


FIG. 132.—*Silicate melting curves*

The ordinates represent temperature differences from an arbitrary reference point

so that the material will easily superheat as well as supercool. Both the melting and freezing curves may be so oblique that no halt in the curves is perceptible. Fig. 132, *C*, shows a heating curve for a slow-melting silicate, which gives practically no information as to the true melting point. Silicates of this class may be differentiated by studying the melting curves obtained for different rates of heating. If the faint trace of a halt in the melting curve shifts with rate of heating, it is fairly conclusive evidence that this method is not applicable. A method which has been employed for such materials by the Geophysical Laboratory is to heat the substance for a considerable time at various constant temperatures and, after quenching in mercury, investigate the crystalline properties under a petrographic microscope. The crystalline structure is destroyed by the melting. Materials of this type are albite $NaAlSi_3O_8$, melting below 1200° C, orthoclase $KAl\ Si_3O_8$, and quartz SiO_2 melting at 1710° C.

The apparatus used by the Geophysical Laboratory for silicate work is shown in Fig. 133. The marquardt or sillimanite porcelain tube *A* is joined to a platinum sleeve *B*, 5 by 1.2 cm, into which tightly fits the platinum crucible *C*, containing the charge. The platinum thermocouple, carefully centered, is inserted with no protection. Great care must be taken to avoid the presence of impurities, such as chips of porcelain from the tube *A*, etc., as the lowering of the freezing point by impurities is proportional to the square of the absolute temperature and becomes very serious at high temperatures. Fig. 132, *B*, shows a heating curve of a silicate containing an impurity.

FIG. 133.—*Silicate melting-point apparatus*

White has found that in general the electrical leakage through the molten salt is not of serious importance. However, this must be considered for precision work at high temperatures, as well as the leakage from the furnace heating current to the couple. The latter may be diminished by preventing any part of the heated couple or crucible from coming into contact with the furnace wall. Thermionic leakage to the couple may be minimized by electrostatic shielding. Leakage may be usually detected by moving the couple and by reversing or cutting off temporarily the heating current.

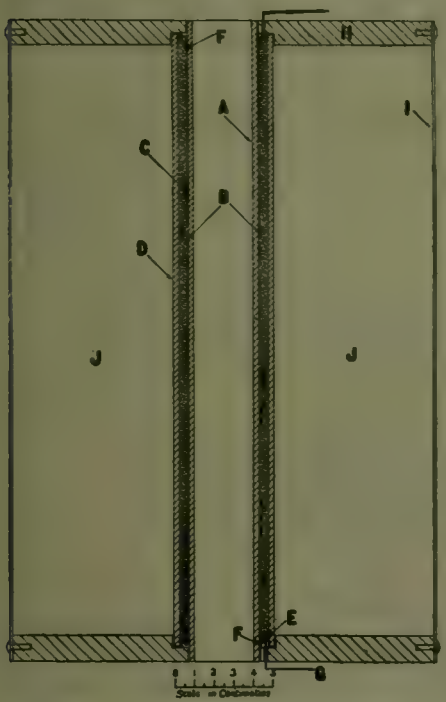


FIG. 134.—*Platinum wound melting-point furnace for silicates and metals*

The rate of heating should be from 2° to 8° C per minute. On account of the tendency of the melting curves to obliquity and on account of the small heat transformations involved, it is necessary to maintain a careful furnace control, so that irregularities in heating will not appear on the melting curve. Even then it is possible

that more sensitive methods of thermal analysis may be desirable, such as the inverse rate or the differential methods described elsewhere.[30].

Since an oxidizing atmosphere is necessary, the furnace employed for high melting-point work with salts and silicates must be wound with platinum. Fig. 134 shows a furnace suitable for temperatures up to 1550° C. The tube *A* of Norton alundum, R. A. 98, 33 cm long, 3 cm bore, and 3 mm wall, is wound with platinum or platinum-rhodium ribbon *B*, 1 to 2 cm wide and 0.01 or 0.02 mm thick, with the turns about 3 mm apart. The ends of the foil are bound with a turn of platinum wire *F*, to which are fused the heavy-silver lead wires *G*. Around the tube *A* is a second tube *D*, and the space between the two tubes is packed with Norton R. R. alundum, 120 mesh, to prevent local heating of the ribbon. The space between the shell *I* and the tube *D* is filled with sintered magnesite.

(1) *Table of Melting Points.*—The following table gives the melting points of several salts and silicates determined at the Geophysical Laboratory:

TABLE 25.—Melting Points of Salts, Silicates, and Other Compounds

Substance	Formula	Melting point
		°C
Sodium molybdate	Na_2MoO_4	687
Borax	$Na_2B_4O_7$	741
Sodium chloride	NaCl	801
Sodium sulphate	Na_2SO_4	884
Sodium metasilicate	Na_2SiO_3	1088
Lithium metasilicate	Li_2SiO_3	1202
Lithium orthosilicate	Li_4SiO_4	1255
Eutectic between diopside and calcium metasilicate	$MgSiO_3.CaSiO_3$ 60 per cent, $CaSiO_3$ 40 per cent	1357
Penta calcium trialuminate	$5\ CaO.3Al_2O_3$	1382
Diopside	$MgSiO_3.CaSiO_3$	1391
Eutectic between calcium metasilicate and cristobalite	$CaSiO_3$ 77 per cent, SiO_2 23 per cent	1426
Calcium metasilicate	$CaSiO_3$	1540
Anorthite	$CaSiO_3.Al_2SiO_5$	1551
Magnesium metasilicate	$MgSiO_3$	1554
Calcium aluminate	$CaO.Al_2O_3$	1592
Cristobalite	SiO_2	[a] 1710

[a] Quenching method.

(*d*) Technical Melting-Point Determinations.—For ordinary technical testing, in which an accuracy of 5 to 10° C is sufficient, the general procedure is the same as that discussed for pre-

[30] For further details on silicates see publications of the Geophysical Laboratory in the Am. J. Sci., and Jaeger, Physico-Chemical Measurements at High Temperatures, Walters, Groningin, Holland; 1913.

cision work, except that less care is required in experimental details. When large couples are used it is necessary to procure much larger crucibles, approximately in the same proportion illustrated by Table 23. It is not a wise practice to immerse a bare base-metal couple directly in a molten salt or metal. The couple should be protected by a metal or ceramic tube, preferably of thin wall, in order to reduce heat conduction. For metals, Dixon graphite crucibles, which are less susceptible to oxidation than the Acheson graphite, or pure fire clay (except for aluminum) are satisfactory; while iron, calorized iron, nickel, nickel-chromium alloys, or duriron are serviceable for salts. For the checking of thermocouples pots of commercial tin, lead, zinc, sodium chloride, and copper of a fair degree of purity are sufficient. The melting points of these materials will rarely differ from those of the pure substances by 10° C, and, if close checks are required, the melting points may be determined by means of an accurately calibrated couple. A gas furnace may be employed for heating, but the flame should not be allowed to strike the crucible on one side only. If the burner is directed along a radius of the furnace instead of tangential to its surface a baffle may be interposed before the crucible, or the crucible may be mounted on a pedestal above the flame. Under these circumstances it is desirable to protect the crucible by an outer metal tube. An excellent form of gas furnace is the melter's furnace illustrated by Fig. 135. For ordinary laboratory work size No. 3 is suitable. The furnace is provided with three burners *D*, only one of which appears in the drawing, mounted tangentially to the circumference in order to produce a spiraling flame. Excellent temperature uniformity is thus obtained.

(*e*) Wire Method.—The wire method of determining melting points of metals consists in inserting at the hot junction of the couple a small length of wire drawn from the material to be investigated. Usually a rare-metal couple is employed. The couple is cut apart at the hot junction and the sample wire, preferably of about the same diameter as the wire of the couple and about 1 cm or less in length, is fused (not soldered) in. The couple is then mounted in a narrow tube, which may be closed at the lower end, and inserted into a uniformly heated furnace, as illustrated by Fig. 136. The temperature of the furnace is increased very slowly as the melting point is approached, and the emf of the couple is observed when it comes to a halt during melting. This occurs

slightly in advance of the instant that the circuit is broken by the melting. The temperature corresponding to this emf on the calibration curve of the couple is the desired melting point. The introduction of the short piece of metal produces no effect on the thermoelectric circuit, provided it is at a uniform temperature. The two metals most satisfactorily employed with this method are gold and palladium, although many other metals could be used with the proper atmosphere. The experimental

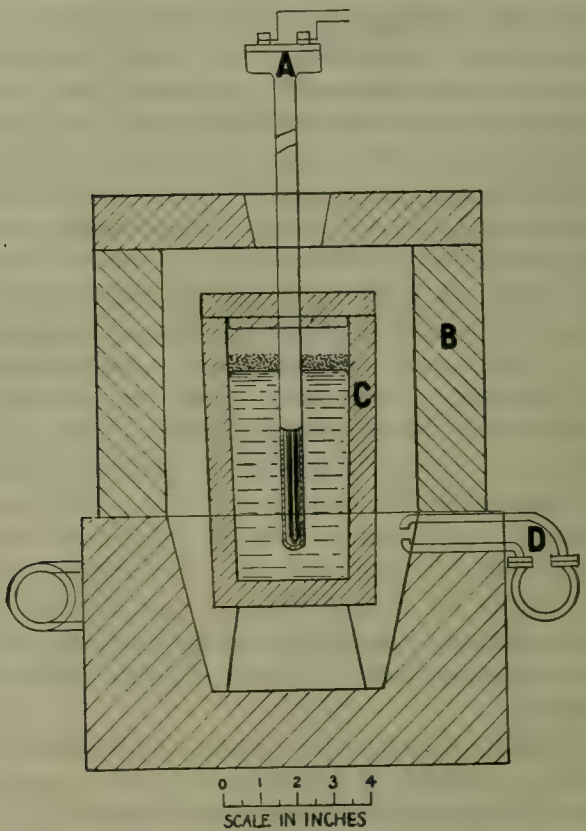


FIG. 135.—*Gas furnace for technical melting points*

manipulation for palladium is a little difficult. A platinum-wound furnace similar to Fig. 134 must be used, and the two insulating tubes of the couple must not come into contact with each other or with the furnace walls; otherwise electrical leakage occurs. A leak between the wires of the couple produces a peculiar effect at the instant of melting of the palladium. Instead of the galvanometer showing zero deflection, it will indicate the very large voltaic emf developed when the porcelain acts as an electrolyte,

since the metallic shunt of low resistance across this emf is broken when the palladium melts. With proper care results by this method are reproducible to 1° C. It is frequently convenient to sight a microscope of low power on the loop of wire and observe the melting. If the rate of heating is slow, sufficient time elapses from the incipient melting to secure several reliable potentiometric readings before the circuit is broken.

The wire method is occasionally employed for copper heated in air. The temperature so obtained however, is not reliable. It may be any value from 1063 to 1083° C, depending upon the rate of heating, and hence upon the amount of oxide formed. With very slow heating the wire may be converted into CuO_2, which melts at about 1190° C.

FIG. 136.—*Apparatus for wire method*

3. USE OF RESISTANCE THERMOMETERS

The standard temperature scale as used and distributed by the Bureau of Standards is defined in the interval −40 to 450° C by means of platinum resistance thermometers calibrated in ice, steam, and sulphur vapor (444.6° C). Platinum of a high degree of purity is readily obtainable, and its purity is assured if the mean temperature coefficient of resistance of the thermometer between 0 and 100° C is not less than 0.00388, and if the constant δ of the Callendar equation used for expressing the relation between temperature and resistance is not greater than 1.52. The temperature scale from 450 to 1100° C is defined by the melting points of pure antimony, silver, gold, and copper, and interpolation between these points is based on the temperature scale defined by the rare metal couple (Pt, 90 per cent Pt−10 per cent Rh) calibrated at two of these fixed points, antimony and copper, and a third point, zinc, fixed by the resistance thermometer.

It has been found that the scale defined by the resistance thermometer calibrated as above agrees with the thermocouple scale as closely as this may be reproduced, even up to 800° C. Above this range the behavior of a resistance thermometer is likely to be erratic. The calibration is subject to changes on account of volatilization of the platinum and strains introduced by the warp-

ing of the mica frame upon which the wire is wound. Reliable precise measurements can be obtained only by intercomparison of several thermometers. Below 800° C, however, the temperature scale defined by the resistance thermometer is probably more closely reproduced by different resistance thermometers than is the thermocouple scale reproduced by different thermocouples. Hence when very high precision is required, 0.1° C or better, the resistance thermometer offers some advantages in the range 450 to 800° C. Below 450° C the resistance thermometer is the most accurate means of temperature measurements, since its temperature scale is accepted as standard. In melting-point work of the highest precision from 450 to 500° C there is some question as to whether the error resulting in a temperature gradient over the entire bulb of a resistance thermometer introduces as large an error as may arise with a thermocouple, which requires a much smaller region of temperature uniformity; for example, the error due to inhomogeneity of the wires of the couple.

The general technique to be observed in melting-point determinations with the resistance thermometer is the same as that required in thermocouple work of the highest precision. The large size of crucibles described in Table 23 is necessary, since the bulb of the thermometer is from 5 to 8 cm long. The most extensive paper on the application of resistance thermometers to melting-point determinations is by Waidner and Burgess.[31] This paper contains a complete bibliography up to 1909. Later developments have been made in only the methods of measurement, such as bridges, etc., and are described by Waidner, Dickinson, Mueller, and Harper and by Mueller.[32]

4. USE OF OPTICAL PYROMETERS

(*a*) METALS AND ALLOYS.—Above 1400° C the difficulties involved in the use of thermocouples for melting-point determinations are very great. Refractory tubes are attacked by the metals and are permeable to gases, which contaminate the couple. The calibration of the couple changes, inhomogeneity develops, and troubles arise from electrical leakage. For these reasons the use of optical pyrometers is desirable and, above 1550° C, necessary. The disappearing-filament type of optical pyrometer is most satisfactory, since it is capable of high precision and requires a very

[31] Waidner and Burgess, B. S. Sci. Papers, No. 124.

[32] Waidner, Dickinson, Mueller, and Harper, A Wheatstone Bridge for Resistance Thermometry, B. S. Sci. Papers, No. 241. Mueller, Wheatstone Bridges and Some Accessory Apparatus for Resistance Thermometry, B. S. Sci. Papers, No. 288.

small source upon which to sight. Special care must be given in the use of optical pyrometers to insure that black-body conditions are closely approximated.

For metals and alloys melting below 1750° C the experimental device shown in Fig. 137 is suitable. The outer tube is made of sillimanite porcelain, melting at 1810° C, and constitutes the unit to be inserted into a furnace. The crucible inside this tube is about 18 mm in diameter and 20 mm high, and is provided with a cover having two holes, 2 to 3 mm in diameter, in the positions illustrated. The pyrometer is focused on the outer hole and the central hole is for observing the progress of a freezing or melting. If the metal in the crucible is molten the central hole appears quite dark against a brighter background because the level metal surface reflects the image of the upper and cold part of the furnace. The surface under the side hole, however, is convex on account of the surface tension of the metal, and the V-shaped depression formed between the metal and the side wall affords a satisfactory black body upon which to sight the pyrometer. The outer tube should be uniformly heated for a distance at least twice its diameter, and the short crucible should be filled about half full with metal. Tall crucibles are not desirable, as they are less likely to be uniformly heated, and any non-uniformity disturbs the black-body conditions. Although it is realized that such a system is not ideal for producing black-body radiation, especially when the temperature is continually changing, as is necessary in melting-point determinations, still it may be concluded on the basis of many careful observations that the error in general does not amount to more than 2 or 3° C. For more precise work it is possible to fix to the cover of the crucible a reentrant tube immersed directly in the metal, and into which the pyrometer is sighted. This method is frequently employed for the standardization of an optical pyrometer by direct melting point determinations.[33]

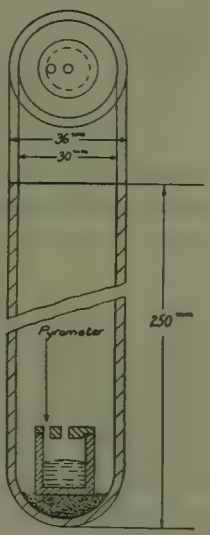


FIG. 137.—*Apparatus for melting points of metals and alloys with optical pyrometer*

[33] Kanolt, B. S. Tech. Papers No. 10. Foote, Met. and Chem. Eng., 11, p. 97, 1913.

(1) *Table of Melting Points.*—The following table gives the melting points of several pure metals, which with their alloys may be investigated by the above method:

TABLE 26.—Melting Points from 1400 to 1800° C

Metal	Melting point	Crucible material	Atmosphere
Silicon	1420	MgO, Al_2O_3, or mixtures	Vacuum, N_2, H_2.
Nickel	1452	do	Vacuum, N_2.
Cobalt	1480	do	Vacuum.
Iron	1530	do	Do.
Palladium	1550	do	Air.
Chromium	1615	do	Vacuum, N_2.
Platinum	1755	do	Air.

(2) *Crucibles.*—Crucibles containing either graphite or silica should not be used for precision work with these metals and their alloys. Silica-free crucibles are not sold by the porcelain manufacturers, but can be made in the laboratory without much difficulty from magnesia, alumina, or mixtures of the two. Pure magnesium aluminate does not begin to melt below 1925° C, and hence is sufficiently refractory for the above metals. Pure fused alumina and pure fused magnesia are ground to 150 to 200 mesh and mixed in the approximate proportion 75 per cent alumina and 25 per cent magnesia (the exact proportion is immaterial). The powder is slightly moistened and compressed in a briquetting mold, similar to that illustrated by Fig. 138, into a cylinder having the outside diameter of the crucible. This cylinder is fired in a graphite crucible at a temperature of 1700° C for a few moments. The Arsem furnace, described later, or any carbon-resistor furnace having a clean atmosphere and capable of maintaining this temperature, is satisfactory. The cylinder sinters to a hardness sufficiently strong to be handled, and with care it is possible to drill out a hole large enough for the metal with an ordinary twist drill. The crucible is then refired at a temperature between 1800 and 1900° C. A cover is readily made in the same manner.

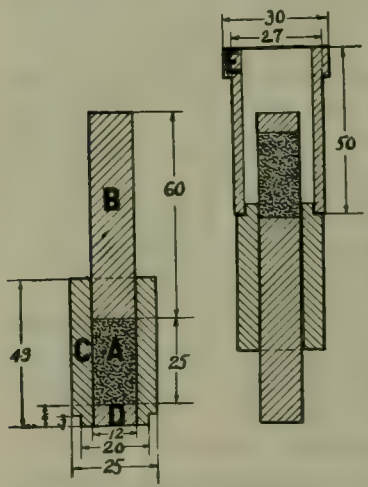


FIG. 138.—*Briquetting mold for refractory materials (dimensions in mm)*

For ordinary testing with metals other than palladium and platinum, the sillimanite crucible ("impervite" or "usalite") may be employed. These are, of course, attacked and must be discarded after each determination, but experiments indicate that the impurity introduced in the metal by a single heating affects the melting point less than 10° C. Such crucibles are useful for ordinary work with steels and nickel-chromium alloys.

(3) *Furnaces.*—None of the metals or alloys of high melting point, except some of the rare metals, can be melted in air without serious oxidation. The use of a vacuum furnace, the residual gases being CO and N_2, is satisfactory except for palladium and platinum. Fig. 139 illustrates such a furnace designed by Arsem.[34] It is readily and accurately controllable and a clear atmosphere is assured. The graphite spiral *C* is mounted in water-cooled copper electrodes. *F* is a radiation shield of graphite filled with magnesia. The pyrometer is sighted through the glass window *D* into the sillimanite tube *B* (see also Fig. 137), which is suspended from the top of the furnace. In the place of *A* the crucible shown in Fig. 137 is mounted on a bed of alundum. The porcelain tube is fairly impermeable to the hot gases and carbon vapor. With the first heating in a vacuum furnace the liquid metal usually sputters on account of evolution of gases, and may throw off the cover of the crucible, but after the first melting satisfactory readings can be taken. Suitable rates of heating or cooling near the melting point are from 10 to 20° C per minute. In this range temperature measurements should be made very frequently—every 10 seconds, if possible. Fig. 140 illustrates typical freezing curves for pure iron and chrome steel by this method.

For temperatures below 1550° C the sillimanite tube of Fig. 137 may be heated in a platinum-wound furnace, if desired. The proper atmosphere for the metal can be obtained by fitting the sillimanite tube with a metal top containing a window and two side tubes through which N_2 is drawn. Another method is to insert a long porcelain tube through the furnace, in the center of which is mounted the crucible. Gas is led in at the top of this tube and out at the bottom.

Crucible melts of palladium and platinum are seldom made. Both of these metals must be melted in an oxidizing atmosphere, since in a reducing atmosphere the slightest trace of silica, which is present in nearly every type of furnace, is disastrous. Palla-

[34] Arsem, J. Am. Chem. Soc., 28, p. 921; 1906. Kanolt, B. S. Tech. Papers No. 10; B. S. Sci. Papers, No. 212.

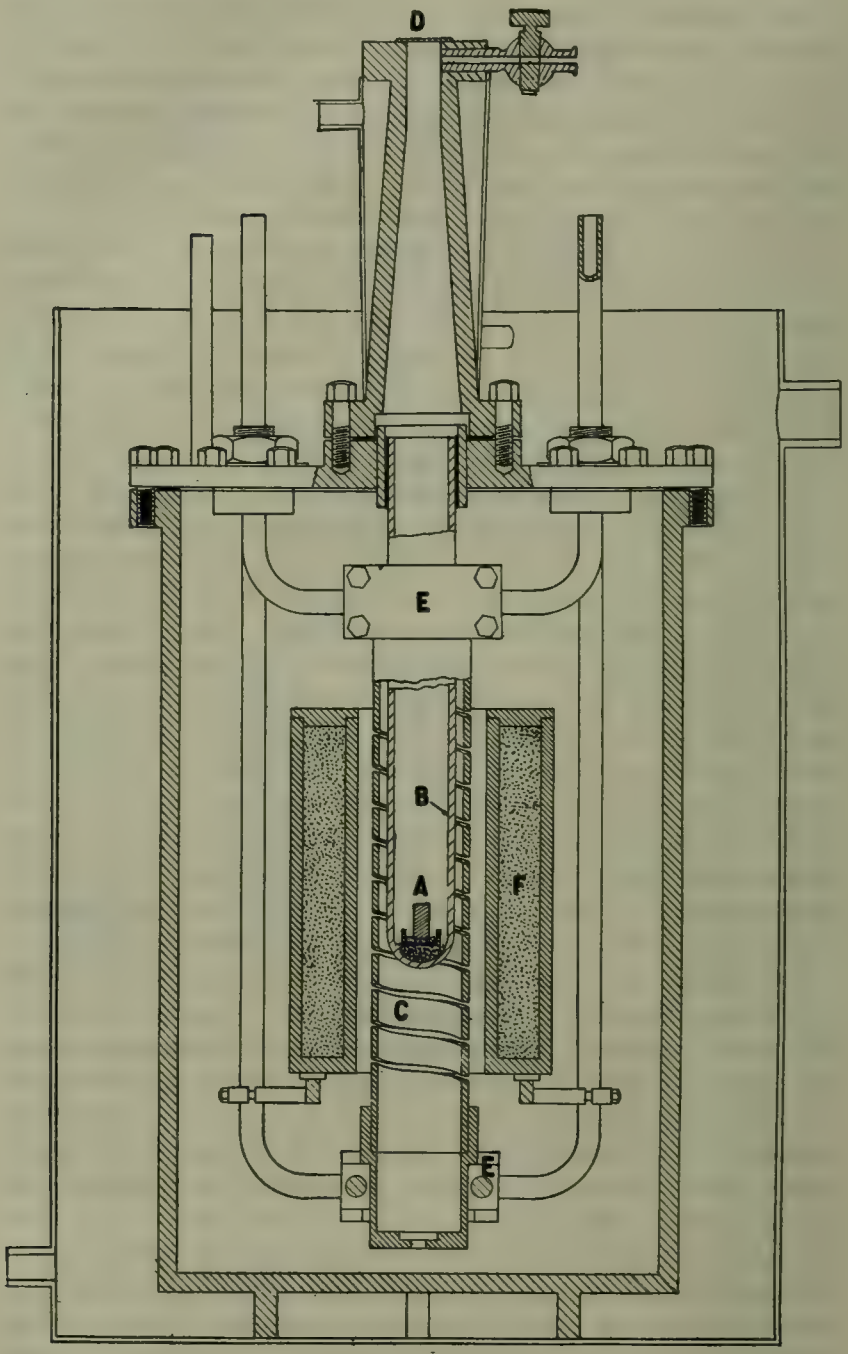


FIG. 139.—*Graphite resistance vacuum furnace*

dium may be heated in a lime, alumina, magnesia, or magnesium-aluminate crucible in a platinum-wound furnace. Platinum could be melted in a furnace wound with a platinum-rhodium alloy, but the method has never been attempted. In several cases a furnace consisting of an iridium tube in which the tube serves as the resistor element has been used. The method is open to serious objections, however, on account of the rapid volatilization of

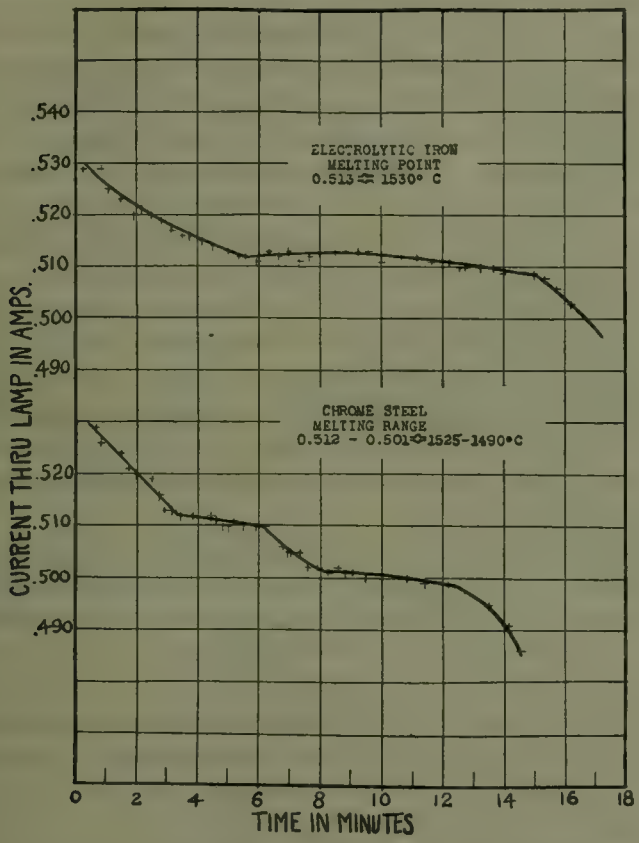


FIG. 140.—*Freezing curves of pure iron and chrome steel*

iridium even when the material is coated with a protective paint, such as a refractory earth mixture.[35]

A furnace recently developed by Northrup [36] heats any electrically conducting material by inducing eddy currents in it. This method is admirably adapted to the melting of easily contaminated metals. The heating may be made with any desired

[35] Waidner and Burgess, B. S. Sci. Papers, No. 55.

[36] Northrup-Ajax High-Frequency Induction Furnace, Chem. Met. Eng.; Aug. 1, 1918. Principles of Induction Heating with High-Frequency Currents, Trans. Am. Electrochem. Soc.; 1919.

gas as an atmosphere or in vacuum. Limitations upon proper refractory containers and insulators are reduced to a minimum. It is even unnecessary to use silica-free crucibles for metals such as palladium and platinum. Temperature uniformity through the charge is secured, thus permitting satisfactory measurements with the optical pyrometer. The Northrup furnace operates on an entirely new principle and its many possible applications are still being developed.

(4) *Other Methods.*—For metals melting much above the range of platinum a modification of the wire method may be used. The material in the form of wire or ribbon is mounted as a filament of an electric lamp and is gradually heated electrically until it melts. The apparent or brightness temperature is measured by an optical pyrometer at the instant of melting. In order to correct the observed temperature to true temperature the value of the emissivity of the material must be known. This involves a separate and difficult experiment. In some cases the material in sheet form is folded into a wedge and electrically heated, and the pyrometer is sighted into the opening of the wedge, the radiation from which is approximately black. Various other methods are employed, such as sighting on a bead of the molten metal in an electric arc, extrapolation of the temperature-current curve for a lamp filament, etc.[37]

(5) *Micropyrometer.*—In order to determine the melting points of microscopic samples of materials, Joly devised the meldometer. This consists of an electrically heated platinum strip the linear expansion of which is a measure of its temperature. The relation between elongation and temperature is determined by placing samples of pure materials on the strip and observing the melting point through a microscope while the elongation is measured by a screw adjustment. A more satisfactory method is by use of the Burgess[38] micropyrometer, which operates on the same principle as the disappearing-filament pyrometer, except that the lamp is mounted in a microscope instead of a telescope. (See Fig. 141.) This is sighted on a platinum strip and on the strip in the field of view is placed a sample of the material to be melted, usually from 0.1 to 0.001 mg in weight. The strip is inclosed in a water-cooled container having a clear-glass window and a re-

[37] Waidner and Burgess, B. S. Sci. Papers Nos. 40 and 55; J. d. Phys. **6**, p. 830; 1907. Forsythe, Astrophys. J., **12**, p. 364; 1911. Mendenhall and Ingersoll, Phys. Rev., **25**, p. 1; 1907. Pirani and Meyer, Verh. d. Phys. Ges., **14**, p. 426; 1912. Wartenberg, Verh. d. Phys. Ges., **12**, p. 120; 1909. Langmuir, Phys. Rev., **6**, p. 152; 1915. Worthing, Phys. Rev. **10**, p. 377; 1917.

[38] B. S. Sci. Papers Nos. 198, 205, and 242.

ducing or neutral atmosphere is maintained. The temperature of the platinum strip is measured at the instant of melting, which is indicated with unmistakable evidence, since the material either gathers into a ball or spreads out on the strip. The pyrometer is calibrated by employing metals of known melting points. A natural question which arises is whether the sample actually takes up the temperature of the strip. If the sample were heated by radiation alone, since it receives radiation from a solid angle 2π and radiates through a solid angle 4π, it can be shown on the basis of the Stefan-Boltzmann law that the absolute temperature of the sample would be roughly only 0.8 that of the strip. On the other hand, the microscopic sample acts as a shunt on the platinum strip, and more heat may be developed at this point, tending to increase the temperature. Thermal conduction along the strip reduces this effect. The fact that samples of different sizes appear to give the same value for the melting point indicates that all these effects tending to produce temperature non-uniformity are small and that the sample actually assumes the temperature of the strip, the heat conduction and gas convection compensating for the deficiency in the direct radiation. The method must be used with caution, however, when applied to other materials than metals, especially in a vacuum. It has been found that errors of 50° or more resulted when gold was heated on a graphite strip in vacuum.

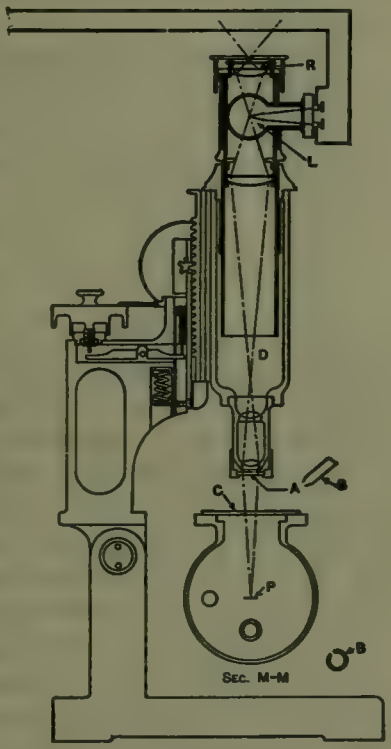


FIG. 141.—*Micropyrometer*

(*b*) REFRACTORY MATERIALS—(1) *Refractory oxides.*—Refractory oxides are the basic constituents of minerals and of ceramic refractory materials. Their melting points are very high, and it is necessary to observe special precautions to avoid contamination and to eliminate the smoke produced by the vaporization of the

oxide. The Arsem furnace shown in Fig. 139 was used by Kanolt [39] for determining the melting points of magnesia, lime, alumina, and chromium oxide. In each case heating curves by the temperature-time method were obtained with an optical pyrometer, and the results were confirmed by examination of the materials heated above and below the observed melting points. The rate of heating was about 100° C per minute near the melting point.

The following table of melting points is a summary of Kanolt's work:

TABLE 27.—Melting Points of Refractory Oxides

Oxide	M. P. ° C	Crucible material	Atmosphere
MgO	2800	Graphite	Stream of CO and N_2 at atmospheric pressure
CaO	2570	Tungsten, CaO	Stream of H_2 at atmospheric pressure
Al_2O_3	2050	Tungsten, graphite	CO and N_2 vacuum; stream of CO and N_2 at atmospheric pressure
Cr_2O_3	1990	do	CO and N_2 vacuum

Magnesia was melted in a special form of graphite crucible, Fig. 142, which was mounted in the Arsem furnace. The pyrometer was sighted through the tube *B*, on the bottom of the tube *A*, immersed in the magnesia. *CO* and N_2 at atmospheric pressure were passed through *BCD*, thus clearing the field of view from smoke. Lime was melted in a similar container, except that the parts in contact with the lime were made of tungsten. Fig. 143 shows another method employed for lime. The tube *O*, compressed from pure lime, was suspended in the furnace, and a stream of hydrogen was passed in at *P* and out at *Q*, to avoid the presence of smoke. Alumina was melted in vacuo in the same form of crucible used for magnesia and also in a graphite crucible, shown by Fig. 144. This latter method with crucibles of both graphite and tungsten was employed for chromium oxide.

(2) *Fire Bricks and Ceramic Materials.*[40]—Refractory materials such as fire bricks, fire clays, and minerals are composed of compounds of the refractory metallic oxides, solid solutions of the oxides, pure oxides, and mixtures of these classes, together with a variety of chemical compounds. The oxides, silica, and alumina occur most commonly in refractory materials, and frequently in combination with these are the oxides of the alkaline-earth, the alkali, the iron group, and the rare-earth metals.

[39] Kanolt, B. S. Tech. Papers, No. 10; B. S. Sci. Papers No. 212.

[40] For a more detailed discussion see L. I. Dana, Bull. Am. Inst. Min. and Met. Eng.; Sept., 1919.

(3) *Meaning of the Melting Point.*—In the case of refractory materials, which are either amorphous or heterogeneous mixtures of oxides and other substances, there is no definite melting point. The change from the solid state to one in which the material will flow is gradual over both a temperature and time interval. In addition, physical and chemical reactions which are not equilibrium reactions often take place during melting. These phenomena practically prohibit the use of melting curves and, for ordinary technical purposes, the criterion of marked flow, although indefinite from the pure scientific standpoint, is adopted. Accordingly, the practical definition of the melting point of a refractory material is that temperature at which under specified conditions a marked and distinct flow of the material begins. In terms of the deformation of a cone or cylinder the melting point is midway between the temperature at which the deformation begins and the temperature at which the material fuses into a lump or ball, or is completely bent over. The softening or fusing point of a refractory should be defined in the same manner, making the three terms synonymous. If the refractory is a pure crystalline compound, it possesses a true melting point, but often, as, for example, with quartz and albite, the change of state takes place so slowly at the melting point that the substance may be superheated. Thus with quartz the viscosity is so high that a marked flow is not perceptible until the material is superheated by 40 or 50° C.[41] For technical testing the criterion of marked flow is usually taken to define the so-called melting point even for pure crystalline compounds, and thus, on the basis of such a definition, the melting point of quartz is about 1750° C.

FIG. 142.—*Arrangement for melting magnesia*

FIG. 143.—*Arrangement for melting lime*

[41] Quartz begins to flow at 1750° C. (Kanolt, B. S. Tech. Papers, No. 10) while its true melting point is 1710° C (Ferguson and Merwin, Am. J. Sci., **46**, p. 1: 1918.]

(4) *Conditions Affecting Observed Values of the Melting Point.*—The results obtained on the basis of the above definition of melting point are influenced by a number of experimental factors as follows: (*a*) Chemical composition; (*b*) size of the particles, and size, shape, and orientation of the sample; (*c*) time and rate of heating; and (*d*) pressure and chemical nature of atmosphere.

The relations between these factors are so complex that in most cases it is not possible to predict the magnitude or sign of the change in melting due to a variation in one or more of the above conditions. It must be noted that the melting point in actual use of the refractory material may differ considerably from that observed in the laboratory on account of the fact that the conditions of use are not reproduced in the laboratory. Thus contamination of the refractory by molten metals, slags, flue dust, etc., may alter the melting point because of the chemical reaction; also, the time and rate of heating may be different. Under load the softening or melting point of a refractory is lowered on account of plastic flow. For this and other reasons the value of the melting point determined in the laboratory is not the only factor, as far as failure is concerned, to be considered in the choice of a refractory for an industrial installation.

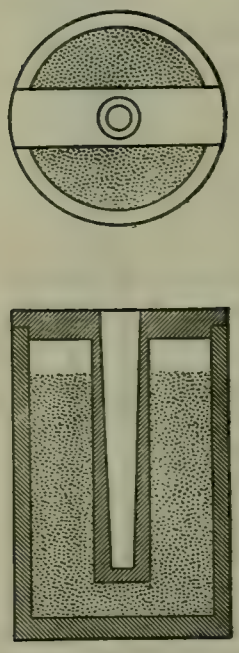


FIG. 144.—*Crucible for melting alumina and chromic oxide*

(5) *Method of Melting-Point Determination—Sampling, Grinding, and Molding.*—Fire brick and other refractories of nonuniform texture and composition or with particles larger than 30-mesh should be carefully sampled. It is accordingly necessary to grind the material and mold the powder into the form of a cylinder or cone. If the refractory is of fine and uniform texture, however, a piece may be chipped off and shaped to the proper form. Fine refractory powders may be briquetted directly. Since the size of the particles may affect the melting point, the degree of fineness should be specified, at least approximately. For a large variety of substances, grinding to pass an 80-mesh screen has proven satisfactory. The material is then slightly moistened and compressed into a cylinder about 2.5 cm high and 1.2 cm in diameter, by means of the brass or polished steel mold shown in Fig. 138. In

this figure *C* is a cylinder, *B* the piston, *D* a plug, and *A* the powder. In order to remove the compressed cylinder, the mold is inverted, and the cylinder *E* is placed on top and the sample is slowly pushed out.

(6) *Time and Rate of Heating.*—The rate of heating from room temperature to about 1000° C for substances melting above 1500° C may be very rapid. A satisfactory total time of heating is from 30 minutes to 2 hours with a rate of from 5 to 10° C per minute from about 50° below the melting point and during melting. Since the observed melting point depends to some extent upon the rate of heating, it is necessary to specify the temperature-time curve in order that results be reproducible. The following table illustrates the manner in which this may be done with sufficient exactness:

Typical Time-Temperature Relation

Temperature interval	Time
	Minutes
Room temperature to 1000° C	20
1000 to 1650°	15
1650 to 1700° M. P.	5

(7) *Type of Furnace and Conditions Existing Therein.*—A furnace for melting refractories should be capable of easily maintaining a temperature of 1800° C, and means must be provided for close temperature regulation. The atmosphere within the furnace must not react appreciably with the specimen; otherwise the material must be protected. Graphite or carbon-tube and spiral-resistance furnaces operated in air oxidize rapidly, but may be somewhat protected by passing a neutral or reducing gas through them. The gas may also serve the purpose of carrying away smoke, so that accurate optical temperature measurements may be made. Crushed carbon, graphite, kryptol, or carbon-plate resistor furnaces do not permit of very precise regulation. Some forms of furnaces are so constructed that it is difficult to use an optical pyrometer. The vacuum furnace of the Arsem type, although requiring greater accessory apparatus, is more desirable on account of its freedom from smoke and because good control is possible at extreme temperatures, even at 2500° C. Furthermore, the atmosphere can be made much less reducing than that in an ordinary graphite furnace. Many refractories are seriously attacked in a carbon and carbon-monoxide reducing atmos-

phere, and it is usually not possible to judge by simple inspection of the melted sample whether the reduction is appreciable. In many cases the surface of the specimen is attacked, and a shell of higher melting point is formed, so that, while the inner uncontaminated material may have been melted, there is no outward evidence of this fact. In general, the sample should be protected from the reducing atmosphere by a refractory tube of low porosity. In the Arsem furnace the sample *A*, Fig. 139, is protected by the sillimanite tube *B*. For temperatures above 1800° C no suitable protection tubes have been made. In certain special cases it may be desirable, if possible, to determine the melting point of a refractory under the atmospheric conditions to which it is subjected in actual use. Indirectly the pressure of the atmosphere may affect the melting point. For example, in a vacuum the more volatile and fusible substances, such as alkalies, may distill, and thus cause a rise of melting point.

There appear to be some possibilities in the development of gas furnaces for high melting-point determinations. With a suitable construction and air pressure of 10 pounds and more, temperatures of 1650° C may be maintained with illuminating gas and 1800° C with natural gas. By recuperating the waste heat or by preheating the gases the above temperature limits may be increased. It should be pointed out that for reliable melting-point work it is necessary to maintain a high temperature, not on a restricted small surface, but throughout a considerable volume.

(8) *Temperature Measurements.*—Pyrometric cones have been employed as a rough measurement of the melting temperature of refractories. The refractory is thus said to have a softening point corresponding to a certain cone number. Seger and similar cones serve a useful purpose under slow rates of heating in measuring heat effects during the firing of ceramic products, but in the laboratory where the rates of heating are comparatively rapid and may vary considerably the determination of the cone softening point is not of much significance. For it has been repeatedly shown by numerous investigators [42] that the softening point of Seger cones depends upon the nature of the atmosphere and especially upon the time and rate of heating. Usually the more rapid the rate of heating the higher the softening temperature, variations of 50° C being easily obtained. In order that the indications by a Seger cone possess reliability, it is necessary to specify

[42] Sosman, Trans. Am. Ceram. Soc., 15, p. 482; 1913.

the atmosphere and the time-temperature curve in the manner illustrated above for refractories. Thus in the ordinary use of Seger cones a variable factor is introduced in the temperature scale defined by the cone numbers, which may have no relation to the properties and characteristics of the refractory being tested. Hence it is essential to use a strictly fixed and reproducible temperature scale. This is best accomplished by means of the disappearing-filament type of optical pyrometer.

A very slight departure from black-body conditions enables one to discern the specimen and to watch the progress of melting. It is important to keep the eye fixed on the sample in order that the melting may be observed with certainty. Sintering and vitrification take place on heating, sometimes producing shrinkage and bending; this must not be confused with the change in shape on melting. As in the case of metals, the pyrometer should be focused on a surface of the sample oblique to the line of sight, but it must be assured that this surface is not reflecting light from a hotter section of the furnace, thus causing too high a reading. After the determination the sample is removed from the furnace, broken apart, and inspected. The inside surface should appear homogeneous and amorphous or glassy. When the sample has been melted in a vacuum furnace, a cellular structure is usually apparent, due to gases evolved during melting.

(*c*) Softening Temperature of Coal Ash.—The clinkering of coal ash is closely related to its softening temperature. Consequently, the determination of this temperature is of considerable practical importance. The Bureau of Mines has made a very complete investigation of the factors and conditions affecting the fusibility of coal ash and have recommended a standard procedure.[43] The results of their investigations will be briefly described.

Coal ash is composed principally of silica, alumina, iron oxides, and lime, together with small amounts of other oxides. The compounds which exert the greatest influence on the melting point are the iron oxides, because they exist in several states of oxidation, depending on the chemical nature of the gases in which the ash is heated. When the ash is heated in air, the iron is oxidized to its highest states of oxidation, Fe_2O_3 and Fe_3O_4. When heated in a very strongly reducing atmosphere, such as exists in a carbon resistance furnace, all the oxides are reduced to metallic iron. The ash in both of these atmospheres has a higher softening point

[43] Fieldner, Hall, and Field, Bureau of Mines Bull. No. 129.

than when it is heated in moderately reducing atmospheres, such as H_2 and H_2O or CO and CO_2. Under these conditions the oxides are reduced to the ferrous state FeO, forming low melting-point ferrocalcic silicates.

It has been found that the iron in the slag or clinker of an ordinary fuel bed exists in the ferrous state. Consequently, it is preferable to heat the ash in a furnace which will reproduce as nearly as possible the conditions in the fuel bed. A carbon-resistance furnace or one with air as the atmosphere is not suitable. A furnace wound with molybdenum with a gas-tight case and equipped with a complicated arrangement for regulating the mixture of H_2 and H_2O has been used; but it is too complex for industrial use. The simplest and most satisfactory furnace has been found to be the gas furnace shown in Figs. 135 and 145, operated with the maximum excess of gas over air, and containing an atmosphere approximately 50 per cent CO and 50 per cent CO_2. Since some samples of ash may melt as high as 1500° C, it may be necessary to mix a small quantity of oxygen with the air to reach this temperature.

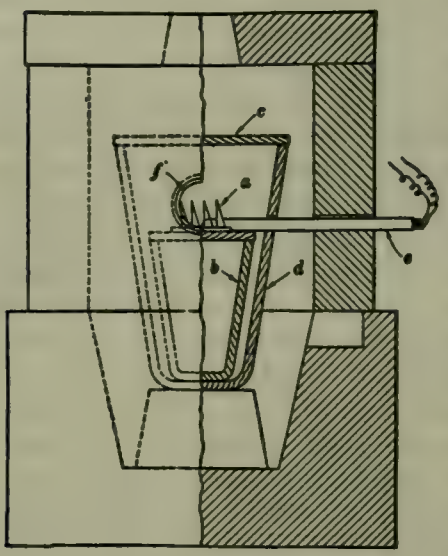


FIG. 145.—*Arrangement for determining fusibility of coal ash*

The following procedure is recommended: After thoroughly roasting the coal the ash is ground to 200 mesh and roasted again in a current of oxygen. Using a 10 per cent dextrin solution as a binder, small triangular pyramids, 20 mm high and 6 mm wide at the base, are made from the powdered ash. These are placed vertically in a muffle or crucible as shown in Fig. 145. A yellow flame burning at least 15 cm above the cover is maintained, and the rate of heating before and during softening should be from 5 to 10° C per minute. The softening temperaure is the point at which the cone fuses down to a lump or ball, which, it will be noticed, is at a further stage of melting than is recommended by the Bureau of Standards for general ceramic material. For the

temperature measurements a thermocouple or optical pyrometer is employed.

(*d*) TABLE OF MELTING POINTS OF REFRACTORY MATERIALS.—The melting points in the following table were determined at the Bureau of Standards, principally by Kanolt, under conditions approximating those described in the section on refractory materials. With the exception of pure kaolin and silica, different samples of the same general type of material may have melting points considerably different from the values tabulated.

TABLE 28.—Melting Points of Common Refractory Materials

Material	Temperature, degrees centigrade	Material	Temperature, degrees centigrade
Fire bricks:		**Clays and sands—Continued.**	
Fire clay	1580–1750	Bauxite clays	1800
Silica	1700–1750	Bauxite Al_2O_3 ($2H_2O$)	1850
Magnesia	2150	Chromite FeO. Cr_2O_3	2180
Bauxite	1600–1800	Silica	1710
Chromite	1850	Silica sands	1700–1750
Clays and sands:		**Miscellaneous:**	
Kaolin, Al_2O_3 $2SiO_2$($2H_2O$)	1740	Coal ash	1100–1500
Fire clays	1400–1740	Alundum cements	1750–2000

XI. STANDARDIZATION OF PYROMETERS

1. STANDARDIZATION OF HIGH-TEMPERATURE THERMOMETERS

High-temperature thermometers may be standardized by comparison with a standard instrument, or the thermometers may be calibrated by immersion in vapors of various boiling liquids. Thus the 100° point may be checked by use of the hypsometer or steam-point apparatus. Other satisfactory temperatures are the boiling points of naphthalene, 218° C, benzophenone 306° C, anthracene 340° C, and sulphur[44] 444.6° C. The boiling-point method, however, is less convenient and in general is more subject to error than the comparison method.

For the range 100 to 320° C the comparator consists of an electrically heated, stirred oil bath, into which the thermometers are immersed. For temperatures up to 550° C an electrically heated lead or tin bath is most satisfactory. The comparator is constructed of iron throughout and contains a stirrer for circulating the molten metal. The thermometers are inserted in thin steel tubes closed at the bottom and immersed in the liquid metal.

[44] See p. 278 for description of the apparatus required

It is not possible to read thermometers totally immersed in the above-described comparators, and hence special attention must be given to the stem corrections. If both the standard and the thermometer under test have the same dimensions and the same number of degrees of mercury column emergent, the stem corrections may be omitted. This is seldom the case, however, and it is usually necessary to compute stem corrections for both thermometers, as described on page 14. Thermometers graduated for short lengths of immersion (e. g., 3 to 5 cm) should not be tested for such immersion on account of the uncertainty as to the temperature of the upper part of the bath. It is preferable to immerse to a greater depth and to correct the observed data for the desired depth of immersion.

The calibration of an improperly annealed thermometer changes with time and use, and even the best constructed thermometers are subject to some variation. If the thermometer is graduated for the ice or steam point, the amount of any change may be detected by check observations in ice or steam directly after a reading at a high temperature. Where no ice-point graduation is provided it is advisable to test the highest point on the scale first. If a preliminary test shows a considerable change in the readings with moderate heating, the thermometer should be annealed in an electrically heated oven as described by Dickinson.[45]

The above brief description of thermometer standardization applies more especially to laboratory thermometers of the etched or inclosed-scale types. In many cases the comparators do not reproduce satisfactorily the conditions to which industrial thermometers are subjected in technical practice; as, for example, the measurement of the temperature of gases or superheated steam. Unfortunately, practically no information is available in regard to the best procedure for such tests.[46]

2. STANDARDIZATION OF THERMOCOUPLES

Thermocouple testing may be divided into two general classes—primary standardization and secondary standardization. In primary standardization the thermocouple is calibrated directly in terms of certain fixed temperatures, defined by the melting or freezing points of a series of chemically pure metals. In a secondary standardization the couple under test is compared with

[45] Dickinson, B. S. Sci. Papers No. 32.

[46] See Wilhelm, Chicago Pyrometry Symposium.

another couple, which has been calibrated as above. Primary standardization is not recommended for most industrial laboratories, as it requires a considerable amount of extra apparatus and laboratory technique. This Bureau undertakes work of such character for a nominal fee and recommends that the industrial laboratory maintain at least two standard rare-metal couples, which may be submitted to the Bureau at intervals for a primary calibration. All the couples are then calibrated in the industrial laboratory by comparison with these standards. However, a number of companies prefer to do their own primary standardization, and for this reason the best methods for primary calibrations as well as secondary calibrations are discussed below.

(*a*) STANDARDIZATION OF RARE-METAL COUPLES.—The rare-metal couple may be purchased completely mounted in a porcelain or quartz tube with the cold junction arranged to operate at room temperature or at the temperature of melting ice. The latter method is preferable for the highest accuracy, but the former method is usually sufficient for most requirements. A suitable ice box is described later.

If desired, the thermocouple wire may be purchased from the platinum refiners and made into couples in the laboratory. One wire should be platinum and the other an alloy of 10 per cent rhodium with platinum, both of the highest possible purity. For general work wires 0.5 mm or 0.6 mm in diameter and 100 cm long serve satisfactorily. A shorter length may be used if the cold junction is operated at room temperature or in connection with the compensating leads described elsewhere.

The hot junction is formed by fusing the two wires in an oxy-gas flame or small electric arc. The wires are threaded through two-hole porcelain insulators and placed inside of a thin-walled protecting tube of quartz or of porcelain glazed on the outside only. The two free ends of the couple are mounted in the ice box and from this point copper leads are carried to the potentiometer.

(1) *Annealing.*—Before standardization the couple should be removed from the mounting and the entire length of wire electrically annealed for a few minutes at about 1500° C. This treatment eliminates the emfs which otherwise would be developed between the hard and soft portions of the wire. Often rare-metal couples contaminated by long-continued use may be restored to their original condition by annealing for an hour or more at this high temperature. Impurities, such as iron oxide, which have accumulated on the surface of the wires may be removed by 50

per cent nitric or hydrochloric acid, or by melting borax on the heated wires and allowing the beads of borax to flow along the wire and drop off. The borax should drop off instead of vaporize in order that it may carry with it any absorbed matter. In case long-continued alternate heating and treatment with borax or acid fail to restore sections of the couple, these badly contaminated portions should be cut out and the couple fused together again with as small a junction as possible. The insulators should be cleaned by heating to incandescence in a gas flame, or, when badly corroded, should be replaced by new tubes.

For annealing electrically the couple is suspended between two binding posts and sufficient current passed through it to raise the temperature of the wire to about 1500° C. A couple made of wires 0.6 mm in diameter requires about 14 amperes, 0.5 mm about 12.5 amperes, and 0.4 mm about 10 amperes.

In general, the contaminated portions do not glow as brightly as the purer sections of the wire. If the wire is not badly contaminated, the darker spots will gradually disappear. If more extreme local treatment is required, the contaminated portions may be wound into a small spiral and the couple heated as before. The coiled portion of the wire loses less heat per unit length by radiation, and hence can be maintained at the desired temperature while the rest of the wire is heated to a dull red. Usually foreign matter produces a gray or dark appearance of the wire when cold, and these contaminated sections are stiffer than the uncontaminated wire.

While couples may be often restored to their original condition by annealing, the disappearance of the gray spots on the wire does not prove absolutely that the couple is free from contamination. The absence of contamination is shown, however, by a homogeneity test. Where high accuracy is required a homogeneity test should be made.

(2) *Homogeneity Test.*—For work of precision each wire of the couple must be uniform in chemical composition and physical condition throughout its entire length. The magnitude of the emf developed by the couple depends upon the composition of the couple in the region of the temperature gradient. The emf developed by an inhomogeneous couple is not necessarily a measure of the temperature of the hot junction. In fact, it is a measure of the temperature of the hot junction only when the region of inhomogeneity lies entirely outside of the region of temperature gra-

dient. Such a fortunate condition would be obtained very rarely in practice.

Suppose, for example, in a furnace 40 cm long, that the temperature is uniform and at 1000° C over the central 20 cm of the furnace and that the temperature drops, linearly with distance, to 0° C over the remaining 10 cm on each side. Omitting consideration of conduction losses, a homogeneous couple would show the true temperature of any desired portion of this furnace, and the temperature indicated would be that corresponding to the position of the hot junction of the couple.

Consider the use of an inhomogeneous couple in which the wires are badly contaminated for a length of 5 cm from the hot junction. This couple would give the correct temperature of the central portion of the furnace when immersed to any depth between 15 and 30 cm, because for such depths of immersion the inhomogeneous portion of the couple lies wholly within the region of temperature uniformity. It would not be possible to measure with this couple the temperature of any portion of the furnace less than 15 cm from the outside, because the region of inhomogeneity would lie in the region of temperature gradient. Under such conditions the couple acts as a compound or differential couple, and it is difficult to predict how much in error its indications may be, since the magnitude of the error depends upon the amount of contamination and the steepness and location of the temperature gradient.

The above example illustrates the fact that the reading of an inhomogeneous couple depends upon the depth of immersion and does not, except by accident, indicate the temperature of the hot junction. It would be impossible to measure, by means of an inhomogeneous couple, the temperature of any portion of a furnace which was nonuniformly heated, unless the couple were calibrated in the position used.[47]

For a homogeneity test a short length of each thermocouple wire is selected as a standard reference specimen. These lengths may be cut from the uncontaminated cold-junction ends of the couple, and one set may be used repeatedly for various couples of the same type. One end of the standard wire—for example, the pure platinum wire—is placed in contact with a section of the corresponding wire of the thermocouple, and the emf is observed between the cold-junction end of the thermocouple wire and the

[47] See section b (4), Checking Thermocouples in Fixed Installations, p. 224.

free end of the standard wire when the point of contact between the standard and the thermocouple is heated to some convenient high temperature, maintained constant.

The point of contact of the two wires is then moved to a different section of the thermocouple wire and the process continued until the platinum wire of the thermocouple is completely explored. The process is repeated for the alloy wire of the couple, except that in this case the specimen of the alloy wire is used as the standard.

If e is the *maximum* difference in microvolts between two readings of emf made along one wire of the thermocouple, and s the value of the thermoelectric power for the couple (micro-

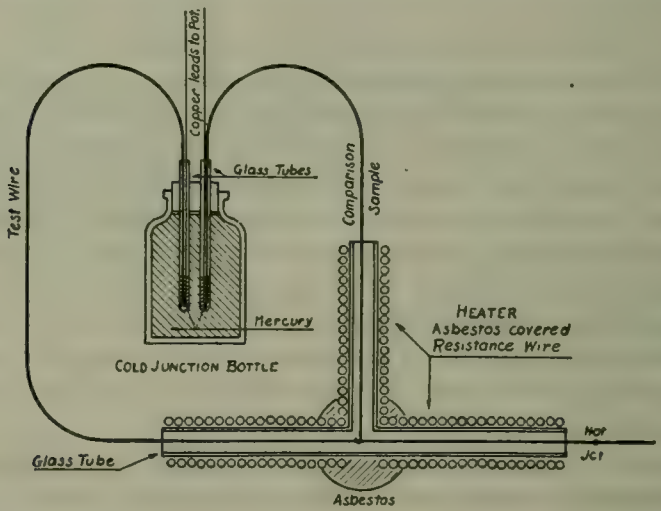


FIG. 146.—*Apparatus for homogeneity tests of rare-metal couples*

volts per degree of temperature), then the *maximum* error usually possible in the use of the couple due to inhomogeneity of this wire is e/s in degrees, provided the temperature of the hot junction in general use does not exceed that employed in the homogeneity test.

If the temperature difference between the hot and cold junctions during the homogeneity test were $t_1 - t_2$, and during the actual use of the couple $t'_1 - t'_2$, the maximum error possible in the use of the couple, due to inhomogeneity of this wire, is usually approximately $\frac{t'_1 - t'_2}{t_1 - t_2} \cdot \frac{e}{s}$ degrees. Similar conditions hold for the other wire of the couple.

The effects of inhomogeneity in both wires may be either subtractive or additive, so that it is evident that to quantitatively correct temperature readings for errors due to inhomogeneity is practically impossible. Merely qualitative results can be obtained from tests of this nature. If the variations in emf observed during the homogeneity test are less than 5 microvolts for $t_1 - t_2 =$ 500° C, the couple can be relied upon for precision work. If certain sections of the couple show larger emf, these sections

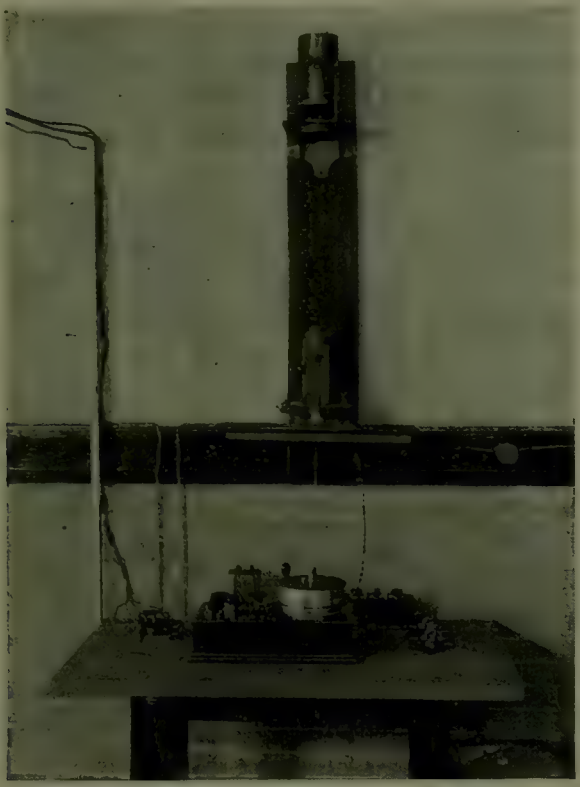


FIG. 147.—*Laboratory potentiometer and accessories*

should be treated by annealing until their condition is satisfactory or should be cut away in case they can not be brought to a proper condition.

The point of contact between the couple wire and the standard wire may be heated by a Bunsen flame, but a more satisfactory method is as follows: The heater, Fig. 146, consists of a pyrex-glass or porcelain T tube having the top and leg of the T wound with resistance wire, through which sufficient current is passed to heat this tube to at least 400° C. One of the wires of the thermo-

couple is threaded through the top of the T and the standard wire is brought into contact with the couple by threading it through the lower part of the T. The end of the standard wire is bent into a small hook, through which the couple passes, sufficient tension being given to the standard wire to secure good electrical contact with the couple. The free ends of the couple wire and the standard are maintained at the same temperature by use of a cold-junction box.

The cold-junction box may consist of a small bottle of mercury, into which dip two small glass tubes closed at the lower end. A few drops of mercury are placed in each tube, and the free end of the couple wire is inserted into one tube and the free end of the standard into the other tube. Copper wires making contact with

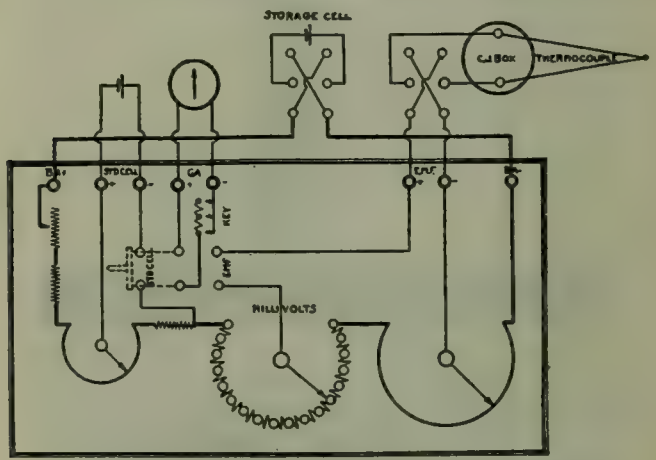


FIG. 148.—*Wiring diagrams of potentiometer installation*

the platinum wires by means of the mercury inside the tubes lead to the millivoltmeter or potentiometer. The copper and platinum must not come in electrical contact except in the mercury. Readings of emf are made for various points of contact along the couple by pulling the couple through the T tube and allowing a minute or so for temperature equilibrium at each new position.

(3) *Laboratory Potentiometer.*—In precision thermocouple calibration a potentiometer especially designed for this type of work should be used. Fig. 147 illustrates a complete potentiometer installation, including galvanometer and reading scale. The wiring diagram is given in Fig. 148.

For work of the highest precision objection has been raised to the use of a potentiometer employing an adjustment of a slide

wire for obtaining either part or all of the emf reading. Instruments are obtainable in which all adjustments are performed by dial settings, the circuits being so arranged that parasitic emfs introduced at moving contacts produce insignificant effects on the measured emf. Examples of this type of instrument are the White and the Diesselhorst potentiometers. Such refinements are, however, seldom essential, except in special research work.

In order to eliminate the effect of accidental thermal emfs developed in the instrument itself, two double-pole, double-throw switches are used, one in the main battery circuit and one in the emf circuit. Readings are made with the two switches thrown first in one direction and then in the other. In one case the parasitic emfs arising in the potentiometer and the galvanometer are additive to that of the couple, and in the other case they are subtractive, whence a mean of the two readings gives the true emf of the thermocouple circuit. In comparing the emfs of two or more couples readings should be taken in the following order and the final means obtained should be used as the calibration data:

Desirable Order for Readings

Position of switches	Couple I	Couple II	Couple III
Normal	1	2	3
Reversed	6	5	4
Normal	7	8	9
Reversed	12	11	10
	Mean emf	Mean emf	Mean emf

Some observers prefer to take all readings with the switches in the normal position. One reversed reading is made and half the difference between a normal and reversed reading is added to or subtracted from the mean of all normal readings. This method is allowable if the parasitic emfs do not vary during the observations.

Care should be taken that the battery switch is in the *normal* position when attempting to balance the instrument against the standard cell. The main battery of the potentiometer should be a 2-volt storage cell of about 50 ampere-hours capacity and should be charged every two months. Details in regard to the use and care of a potentiometer are given in the instruction manual furnished with the instrument.

(4) *Cold-Junction Ice Box.*—A satisfactory ice box is obtained by fitting a small battery jar with a fiber or hard-rubber cover. Through the cover are inserted several small glass tubes closed at

the lower end, two tubes for each couple used. A few drops of mercury are placed in each tube, and the battery jar is kept filled with clean ice shaved or ground into a wet snow.

The cold-junction ends of the couples are inserted, one in each tube, and copper wires lead from the mercury to the potentiometer. The copper wires should be insulated from the couple wires throughout the length of the glass tubes, except where they dip into the mercury. This precaution is very important, for, if the copper and platinum (or alloy wire) are in electrical contact at the top of the glass tubes, the whole value of the ice box is lost.

The glass tubes should be kept clean and dry inside. Moisture is likely to condense in the tube from the atmosphere and should not be allowed to accumulate. A little moisture and dirt at the bottom of the tube will form a galvanic cell giving a large emf. It is desirable, but not necessary, to replace the battery jar by a Dewar flask or thermos bottle having a large mouth, since the ice melts more slowly in this form of container. Fig. 149 shows an ice box suitable for thermocouple work.

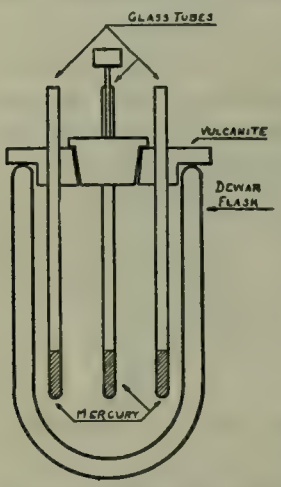


FIG. 149.—*Thermocouple ice box*

(5) *Calibration of Primary Standards.*—The theory and technique of melting-point determinations have been discussed in an earlier chapter. Only the practical application to thermocouple standardization is considered in the following: When a crucible of pure molten metal is allowed to cool, the cooling curve obtained by plotting temperature of the metal versus time is continuous until the freezing point (or transformation point) is reached. During freezing the metal gives out enough internal latent heat to supply the losses due to radiation and convection, and the temperature of the metal remains constant until all the metal has frozen. In this region of the cooling curve the curve is parallel to the time axis. After the metal has frozen it cools in a somewhat similar manner as when completely molten. Fig. 150 shows cooling and heating curves for several metals.

The freezing point of copper (1083° C) is clearly indicated by the flat portion of the second curve. On heating a metal the temperature gradually increases with time until the melting

point is reached. The temperature of the metal then remains constant, as shown in Fig. 150, until all the metal is melted, after which the temperature again increases with time.

The temperatures at which melting and freezing occur are identical for pure metals. When molten antimony is allowed to cool, the temperature usually falls below the freezing point before the metal starts to freeze, a phenomenon known as undercooling. When any of the metal starts to solidify, however, the temperature immediately rises to the freezing point, as shown in Fig. 150, unless the undercooling has proceeded too far. Undercooling may be greatly reduced by stirring the metal or tapping the crucible for an instant just as the freezing point is approached.

Aluminum does not appear to have as well-defined a melting or freezing point as do many other metals. The material is "pasty" over a small temperature range. This may be due to the presence of impurities and is probably not a characteristic of the pure metal.

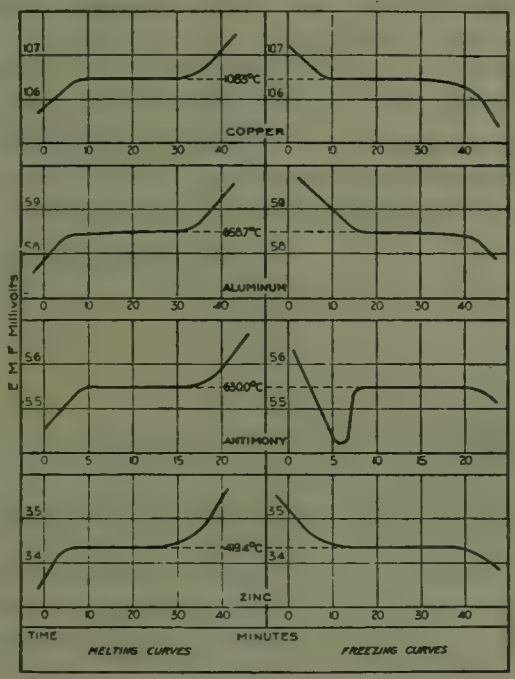


FIG. 150.—*Typical heating and cooling curves*

In the primary calibration of rare-metal couples use is made of the type of cooling and heating curves above described. The couple, properly protected, is immersed in the molten metal, which is then allowed to cool slowly. The emf of the couple is observed as frequently as possible and the time noted for each observation. These values are plotted, and the emf corresponding to the flat portion of the cooling curve is the emf at the freezing point of the metal. A similar process may be carried through by melting the metal.

The temperature versus emf relation for the platinum, platinum-rhodium couple has the following form in the temperature range 300 to 1200° C, $e = a + bt + ct^2$ where e = emf, t = temp. ° C of the hot junction of the couple (cold junction = 0° C), and a, b, and c are constants. Three standard points are required to determine a, b, and c for any particular couple. These fixed temperatures may be the melting or freezing points of zinc 419.4° C, antimony 630° C, or aluminum 658.7° C, and copper 1083° C. The metals must be of the highest possible purity. This is especially the case with antimony, which is very difficult to obtain in sufficient degree of purity. The Bureau of Standards furnishes standard samples of zinc, aluminum, and copper (also tin and lead; see Circular No. 66) with certified melting points. Aluminum of the highest obtainable purity is recommended in preference to antimony for the industrial laboratory. The following shows the method of obtaining the constants a, b, and c:

Observed Calibration Points

Metal	Temperature	emf
	° C	Millivolts
Zinc	419.4	3.436
Aluminum	658.7	5.839
Copper	1083	10.561

Whence, from the above equation, it follows that:

$$3.436 = a + (419.4)\ b + (419.4)^2 c.$$
$$5.839 = a + (658.7)\ b + (658.7)^2 c.$$
$$10.561 = a + (1083)\ \ b + (1083\ )^2 c.$$

These three equations are solved for a, b, c, giving $a = -0.323$, $b = +0.008276$, $c = +0.000001638$. The final equation for this couple thus becomes:

$$e = -0.323 + 0.008276t + 0.000001638\ t^2,$$

where e is the emf in millivolts corresponding to a hot-junction temperature of t° C, and a cold-junction temperature of 0° C. From this equation e may be computed for various values of t° C (for example, 300° C = 2.306 millivolts; 350° C = 2.774; 400° C = 3.249; 1200° C = 11.967, etc.), and a curve or table of e versus t° C may be constructed. Using this curve or table one may readily

find the temperature corresponding to any emf observed in the future use of the couple.

The above equation serves to determine the temperature scale in the interval from 300 to 1200° C. An entirely different equation must be used for points below 300° C. For discussion of this see Burgess and Le Chatelier, "Measurement of High Temperatures." At temperatures higher than 1200° C the following corrections must be added to the temperatures given by the above equation. These corrections apply for all platinum, 90 per cent platinum–10 per cent rhodium thermocouples.

Corrections to Extrapolated Thermocouple Scale

Temperature computed from parabolic equation	Correction to add
° C	° C
1[illegible]	[illegible]
1[illegible]	2
1400	6
1500	14
1600	[illegible]
1[illegible]	39

The standard metal may be used in an Acheson graphite crucible (Dixon graphite is also satisfactory for copper) the inside diameter of which is slightly less at the bottom than at the top. Convenient inside dimensions are about 5 cm diameter and 15 cm deep. For use with the 50 cm^3 metal samples furnished by the Bureau of Standards a crucible 3.5 cm inside diameter and 8 cm deep is recommended. Crucibles should be fitted with a cover having a small hole in the center through which the couple is inserted. The crucible may be heated either in an electric or gas furnace. Fig. 124 shows a convenient form of electric furnace. The crucible containing the metal is shown within a larger crucible and surrounded by graphite. If the furnace is too small for the extra crucible, the graphite may be poured directly into the furnace tube until the crucible is entirely covered. This prevents the oxidation and consequent crumbling away of the crucible.

If a gas furnace is used, the crucible should be placed inside a length of iron pipe, capped at the bottom, and the space between the crucible and the pipe should be filled with powdered or flaked graphite. Instead of an iron pipe a larger crucible of Dixon graphite or fire clay may be used. Fig. 135 shows a suitable gas furnace,

but does not show the iron pipe. In all cases the top of the crucible and the surface of the metal should be covered with 2 or 3 cm of powdered or flaked graphite. This is especially important with copper, as the oxide of copper formed on exposure of the hot metal to the air is soluble in the metal, and thus lowers the melting point to an appreciable extent.

Protecting Tube for Rare-Metal Couple.—The thermocouple must not come in direct contact with the molten metal or metallic vapors. A suitable protecting tube is of porcelain glazed on the outside and closed at the lower end. The diameter of the tube may be 1 cm or less, and the walls should be thin. The couple wires are again protected and insulated from each other by small porcelain insulating tubes or by two-hole porcelain tubing.

It is good practice to remove the couple and insulators from the outside protecting tube when immersing the latter in the molten metal, as frequently the tubes are broken in this operation. This precaution saves the couple in case such an accident takes place. The procedure is, therefore, as follows: Hold the pyrometer tube above the furnace in the heated air until it takes up this temperature. Gradually lower it into the furnace until the top of the crucible is reached. When thoroughly heated, gently immerse it in the metal. Observe that the tube is not broken. If in doubt, insert a small porcelain tube inside the pyrometer tube. If the outer tube is broken, the small tube will be discolored or contaminated with the metal. Another method of detecting a broken tube is to pull it out of the metal, and, if intact, replace it before it has had time to cool. After having determined that the pyrometer tube is good insert the thermocouple. This should be done very slowly, as the rapid insertion of the cold thermocouple may break the protecting tube. After the metal has frozen the protecting tube should be withdrawn, or the furnace should be immediately heated for a melting-point determination. An immersed tube which is allowed to cool with the metal far below the melting point is usually broken. For a small temperature range below the melting point the tube can be readily withdrawn.

Either glazed porcelain or fused quartz (silica) protecting tubes may be used in this work. With aluminum, however, the protecting tube must be itself protected from the metal. This is easily done by making a tubular sheath of Acheson graphite, with walls about 1 mm thick, which slips over the quartz or porcelain tube, as shown in Fig. 123. The porcelain tube need not be removed from the metal before cooling when thus protected. It

is better to have one set of tubes for each metal; but when necessary to use the same tube in various metals this can be done if care is taken to thoroughly clean the tube with nitric and hydrochloric acid. The greatest care should be exercised to see that no dirt of any kind gets inside the pyrometer tubes. Dust or carbon inside the tubes, when heated, forms a reducing atmosphere which

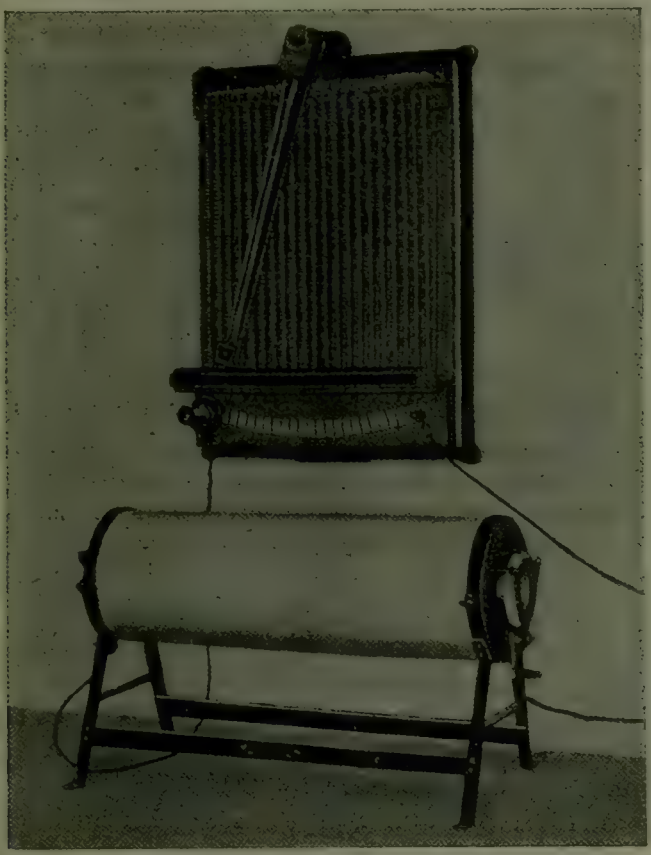


FIG. 151.—*Convenient form of electric furnace for intercomparing rare-metal couples*

acts upon the silica, freeing silicon, which rapidly attacks the platinum.

(6) *Secondary or Comparison Calibration of Rare-Metal Couples.*—The secondary method, as follows, is recommended for industrial laboratories: One, or preferably three, rare-metal couples of wire about 1 meter in length and 0.6 mm in diameter are maintained as primary standards. These couples should be submitted to the Bureau of Standards for test and certification at intervals, depend-

ing upon the amount of use to which they are put, or whenever contamination is suspected. The couples to be tested are compared with these standards. Suitable electric furnaces for such comparison tests are available, designed to operate on either 110 or 220 volts, and may be obtained complete with adjustable rheostat for regulating the current, as shown in Fig. 151. The furnace consists of a porcelain tube wound with platinum foil, surrounded by a larger porcelain tube, around which is packed thermal insulating material. The annular space between the two tubes should be filled with No. 120 mesh Norton R. R. alundum to prevent local heating of the platinum. A convenient size of heating tube is 1 by 18 inches, but larger sizes can be secured if desired. The furnace should be kept clean and should not be used for testing base-metal couples, annealing iron rods, or other processes likely to cause contamination.

The rare-metal couples to be calibrated are removed from the protecting tubes and insulating tubes and annealed, as described previously. The insulating tubes are cleaned, when necessary, by the method described above and then replaced and the couples are tested without the protecting tubes, if the furnace is clean. The hot junctions of all the couples, the standard couple, and the couples under test are connected together with as good thermal contact as possible. The junctions may be fastened together with a small platinum wire and then wrapped with platinum foil. Since the couple wires are thus in electrical contact at the hot junction they must not be allowed to touch one another at any other point. The use of platinum foil around the hot junction reduces temperature gradients to a minimum. If the couples are tested in the protecting tubes this precaution becomes still more important.

The cold junctions of all the couples are maintained, preferably, at 0° C. If three couples are being compared with one standard the readings should be taken in the following order:

Desirable Order for Readings

Reversing switch	Standard	No. 1	No. 2	No. 3
Normal	1	2	8	4
Reversed	8	7	5	
Normal	9	10	11	12
Reversed	16	15	14	13
Means				

The means of the observations on each couple are taken and the temperature is obtained from the reading of the standard couple. Several calibration points are made in this manner in the range 300 to 1200° C. These are plotted, temp. vs. emf, and a smooth curve is drawn through the experimental points for each couple. If it is inconvenient or undesirable to use a cold-junction temperature of 0° C for the couples under test, any other constant temperature may be employed. This can be measured by means of a mercury thermometer. It is nearly as essential to know the temperature of the cold junction as that of the hot junction, since the magnitude of the emf observed depends upon both temperatures.

When a large number of couples must be accurately calibrated, the following method, requiring more elaborate apparatus, is satisfactory. In order to insure equality of temperature between the standard couple and the couple being calibrated without waiting for the furnace to acquire a constant temperature, the hot junctions are fused together. Two separate potentiometers are employed, one connected to each couple. Each potentiometer is provided with a reflecting galvanometer. The two spots of light are reflected onto a single scale, the galvanometers and lamps being so adjusted that the spots coincide with the zero point of the scale on open circuit or when the potentiometers are balanced.

The potentiometer connected to the couple under test is set at an integral number of millivolts, and the temperature of the furnace is increased or decreased until the galvanometer indicates a balance. At this instant the emf, and hence the temperature, of the standard is observed with the other potentiometer. This may be done easily if the spot moves slowly across the scale. By making observation first with a rising and then with a falling temperature, minor errors, such as time lag, are eliminated. Observations are taken at nearly every millivolt for the couple under test, from its lowest to its highest range; the furnace is then cooled and the readings thus repeated in the reverse order. The mean of the observed temperatures corresponding to the various millivoltages gives the final calibration of the couple. For best results about two hours are required, the couple being tested at 10 to 15 points. It is necessary to use a furnace which is not well insulated thermally in order that it may cool sufficiently rapidly.

A form of furnace especially adapted to the rapid testing of a large number of couples consists of a tube of nickel-chromium

alloy (chromel) clamped between two water-cooled terminals. A heavy current from a suitable transformer is passed through the tube, the tube itself serving as the heating element or resistor. For use with base-metal couples a porcelain insulating tube may be placed inside the metal tube, or the base-metal couple may be wound with asbestos cord to electrically insulate it from the walls of the tube. In this case the standard couple, insulated in a nonporous porcelain tube, is placed with its hot junction as near as possible to the hot junction of the base-metal couple. Rare-metal couples may be standardized by taking proper precautions to prevent contamination by the metal vapors from the heating unit. The heating tube of this furnace is exposed to the air, and no thermal insulating material is employed. A large concentric tube may be mounted around the heating tube to reduce the radiation loss, although this is not necessary. A furnace of this type may be heated very rapidly to any temperature up to 1100° C, and it is possible to completely calibrate a couple at five or six different temperatures in a half hour.

(*b*) Standardization of Base-Metal Couples.—It is difficult to secure equality of temperature between the hot junctions of two base-metal couples or between a base-metal and a rare-metal couple. One arrangement for precision calibration is to place the junction of the rare-metal couple in a small saw cut made in the hot junction of the base-metal couple and pinch the jaws of the saw cut until good thermal contact is secured. Small base-metal couples may be fused directly to the rare-metal couple. The rare-metal couple is protected by porcelain tubes to within a few millimeters of the hot junction, and the end of the porcelain tube is sealed to the couple by a small amount of kaolin and water-glass cement or by pyrex glass. This prevents contamination of the rare-metal couple, with the exception of the small length of 2 or 3 mm, which is necessarily exposed to the metal vapors. If the furnace is uniformly heated in this region a small amount of contamination will not cause any error. The method, however, is not feasible for work on a large scale. With fused or squeezed junctions the double potentiometer may be used in calibrating base-metal couples.

(*1*) *Use of a Muffle Furnace.*—Several base-metal couples and the standard rare-metal couples mounted in nonporous porcelain tubes are packed together as closely as possible and tied into a bundle with asbestos cord. The hot junctions are inserted into a length of very heavy iron pipe capped at one end and the open

end from which the couples protrude is tightly packed with asbestos wool. The iron tube and couples are mounted in the furnace and the emf measurements are made as soon as temperature equilibrium is reached. If the muffle furnace is large enough the iron tubes protecting the base-metal couples need not be removed. A satisfactory size of furnace measures 8 by 8 by 30 inches deep. Precision work is impossible in this method of calibration. Even when every precaution is taken to reduce temperature gradients, differences in temperature of 10 or 20° C frequently will be found to exist between the different couples mounted side by side. However, this accuracy is sufficient for many industrial processes.

This method may be greatly improved by employing a large copper block drilled with holes into which the hot junctions of the couples are inserted. The heavy protecting tube is removed from each couple during the test.

(*2*) *Use of Molten-Metal Bath.*—A satisfactory method for calibrating base-metal couples on a large scale is by use of a bath of molten metal. An iron pot about 12 inches in diameter, or larger, and 15 inches deep is filled with tin or lead covered with powdered graphite. This may be heated in a gas furnace or an electric furnace wound with nickel alloy, such as nichrome or chromel. Into the tin bath dip about six iron tubes closed at the lower end and immersed to a depth of 12 inches. The tubes are just large enough to permit easy insertion of the base-metal couple with its iron protecting tube. The standard couple is mounted in one of the six tubes and the base-metal couples are placed in the others. As soon as temperature equilibrium is reached the emfs are measured, that of the standard couple giving the temperature of the bath. A large number of base-metal couples may be tested one after the other at this temperature, and the process is then repeated at another temperature. The tin bath may be used satisfactorily in the range 300 to 1000° C, since tin is a metal of low volatility. Data obtained by the above method are plotted, temperature versus electromotive force, for each couple and a smooth curve drawn representing the calibration. For some types of base-metal couple the curve is slightly S-shaped, showing small inflections near the critical points of the metals of which the couple is constructed. In precision work the calibration must be made at short-temperature intervals over these critical regions to determine the exact form of the temperature-emf curve.

(3) *Single-Point Calibration or Check Point.*—It is frequently desirable to check a base-metal couple at a single point. If the couple is reading emfs, say 2 per cent higher at this temperature than the usual emf of that shown by the original calibration data, it is customary to assume that it reads 2 per cent high at all temperatures. This method of calibration at best is only approximate, and experimentally it is frequently found that a couple reading 2 per cent high at 800° C indicates correctly at 1000° C. However, single-point calibrations are of value, especially as a check upon the behavior of a couple in any particular temperature range.

A convenient standard temperature for this work is the melting or freezing point of sodium chloride (common salt), chemically pure. This material melts or freezes at 801° C. The salt may be used in a duriron crucible heated in a gas or electric furnace. The base-metal couples are immersed without protecting tubes from four to six inches in the bath. Melting or freezing point curves may be observed as already described. These curves are rather oblique on account of the low thermal conductivity of the salt, as discussed in the section on melting-point methods. The exact portion of the curve representing the true freezing point is reached when crystals begin to form along the entire length of the immersed end of the couple. The couple may be used as a stirring rod during the freezing. If the salt has not been previously fused, it must be melted the first time with a cover on the crucible as the imprisoned water in the salt crystals will cause the salt to sputter out badly. After the first melt, however, there will be no trouble in the use of this material. Molten salt reacts somewhat with most base-metal couples, but the reaction is so slow that the couple will not be injured. We have not found any serious change in melting point resulting from heating the salt in contact with iron for several hours.

(4) *Checking Thermocouples in Fixed Installations.*—It is frequently desirable to check the readings of a thermocouple without removing it from the furnace in which it is installed. Tests of this nature are of extreme importance in the technical industries. They are also difficult to perform with precision, and the precision which is obtainable in a laboratory test can not be expected in tests of this nature. However, an ordinary calibration in the laboratory of a base-metal couple which has been in use in a fixed installation for some time, while furnishing results apparently of high precision, does not necessarily hold when the couple is returned to its original installation. This is on account of the heterogeneity developed in a base-metal couple after prolonged heating.

The presence of heterogeneity could be detected by a homogeneity test, but such tests on a large scale are not worth while for base-metal couples except for investigational work and, moreover, do not show how to correct the readings satisfactorily. Such a test would simply indicate that the couple should be thrown away, whereas in its permanent installation it still has a useful life. The throwing away of base-metal couples in a fixed installation as soon as heterogeneity sets up would be a very costly and unnecessary procedure, except in the comparatively rare cases where the highest possible accuracy is required. The degree of heterogeneity developed depends upon the type of installation, depth of immersion, location and character of temperature gradient, etc. These quantities are fairly definite and fixed for any particular installation. If, however, the couple is removed from the installation and calibrated in the laboratory, these quantities are altered and the conditions under which the couple is actually operating can not be reproduced. The calibration of the laboratory may give results quite different from the calibration which would be obtained if it were possible to duplicate exactly all the physical characteristics of the permanent installation. Hence it is evident that if inhomogeneous couples are ever to be used, it is important that they be calibrated or checked in their fixed installation.

The exact method of procedure depends upon the type of installation. A standard checking couple is employed complete with a high-resistance galvanometer, "heat-meter," or portable potentiometer. The hot junction of the checking couple is placed as closely as possible to the hot junction of the couple under test, and the readings of the two couples compared. The main objection to the method is the difficulty in getting the two hot junctions together. One method is to drill a hole in the furnace at the side of each couple permanently installed large enough to permit insertion of the checking couple. The hole is kept plugged, except when comparison tests are being made. The checking couple is immersed in the furnace through this hole to the same depth as the couple under test. The hole should be located as close to the permanently installed couple as possible.

In many installations the base-metal couple and protecting tube are mounted inside another protecting tube of iron, fire clay, carborundum, or some other refractory which is permanently cemented or fastened into the furnace wall. Frequently there is room to insert a small test couple in this outer tube alongside of

the fixed couple. A third method, much less satisfactory, is to wait until the furnace has reached a fairly constant temperature and make observations with the couple under test, then remove this couple from the furnace, and insert to the same depth the checking couple.

If desired, comparisons can be made preferably by either the first or second method at several temperatures, and a curve may be obtained for each permanently installed couple showing the corrections necessary to apply to its readings.

It may be thought that this method of checking couples is unsatisfactory, because in most furnaces used in industrial processes large temperature gradients exist and there is no certainty that the standard couple is at the same temperature as the couple under test. This objection, however, is not serious, because if temperature gradients do exist, in the furnace, of such a magnitude as to cause much difference in temperature between two similarly mounted thermocouples located closely together, the accuracy required by the measuring instruments need not be great, and the reading of the standard couple can be accepted for the standardization of the fixed couple just as satisfactorily as if the true temperature of the fixed couple were known. If temperature gradients exist of such a magnitude that two correctly calibrated couples similarly mounted closely together read, say, 20° C apart, it may be certain that neither couple is registering the temperature of material within the furnace to this degree of accuracy.

(*c*) Cold-Junction Correction.—If a couple is calibrated for a cold-junction temperature of t_o° C and is used with a cold-junction temperature of t_o'°C, the true temperature of the hot junction is that on the calibration plot corresponding to the observed emf e plus the emf e' where e' is the emf developed when the hot junction is at t_o'°C and the cold junction is at t_o°C. The value of e' is positive when $t_o' > t_o$ and negative when $t_o' < t_o$. Frequently the instruments used are graduated in temperature only, in which case to the observed reading is added the quantity $(t_o' - t_o)\ K$, where K is a factor depending upon the couple and the temperatures employed. Values of the factor K for several types of couple are given in Table 9. The factor is obtained from the following equation. This states that

$$K = \frac{\left(\frac{\Delta e}{\Delta t}\right)_{\Delta t_o}}{\left(\frac{de}{dt}\right)_t}$$

is equal to the mean slope of the calibration curve over an interval Δt_o = about 50 °C, in the neighborhood of the cold-junction temperature, divided by the slope at the observed temperature t. The interval Δt_o should embrace any likely cold-junction temperature. Thus, if the calibration were made for a cold-junction temperature of 30° C, the mean slope from 5 to 55° C is the numerator of the above expression. This is obtained by dividing the difference in emfs at 5° C and 55° C, by the difference in temperature, that is, 50° C. If the calibration is made for a cold-junction temperature of 0° C, the mean slope is determined from 0 to 50° C and not from −25 to +25° C, since in practice the cold-junction temperature seldom falls below 0° C. However, since this method is only approximate and since the corrections are always small, the precise manner of determining the slope at the lower limit (that is, the numerator of the correction term) is immaterial.

(*d*) METHOD OF MAKING THERMOCOUPLE JUNCTIONS.—The hot junction of a thermocouple should be made by soldering or fusing the two metals. Silver solder is useful at temperatures below 900° C, or gold below 1000° C, but, in general, the best type of junction is made by fusion. The junction of a rare-metal couple may be made by use of a small oxygen-illuminating-gas flame. Acetylene is not desirable. Special blast lamps suitable for this work are obtainable. The flame should be small, possibly 4 cm long, the tip of the lamp having an opening of from 0.5 to 1 mm. The junction should be fused into a ball about 1 mm in diameter.

Another convenient method of fusing the junction is by use of an electric arc. The couple wires are connected to the positive terminal of the 110-volt direct-current main and the negative terminal is connected through a resistance to a graphite pencil having an insulated handle. The graphite should be about 5 or 6 mm in diameter and should be sharpened to a point. For a couple of 0.6 mm wire a resistance of 30 to 40 ohms is used. Touch together the ends of the two wires of the couple and then bring the point of the graphite pencil into contact with the junction. As soon as an arc is formed draw the pencil away. With a little practice excellent joints can be made in this manner.

This method is also good for repairing broken couples. In this case the junction should be made as nearly as possible of the same size as the wire itself.

Large base-metal couples may be fused either by an electric arc or oxy-gas torch. It is frequently the practice to twist the two wires, as shown in Fig. 6, for a few turns, as this gives greater me-

chanical strength. Before fusing, the junction of the couple is coated thoroughly with borax. This is done by heating the ends of the two wires to a dull red heat and then dipping into the powdered borax. The borax adhering to the wire is then melted and the process continued until a heavy coating of the fused transparent borax is obtained. A stick of borax glass may be used instead of the powder. The couple may be mounted horizontally or suspended with the junction down. The arc or oxy-gas flame is allowed to play upon the junction uutil the metal fuses into a neat bead. If sufficient borax is used, the weld will show no signs of oxidation. The arc employed for this work is of the ordinary type, consisting of two carbons, although for small couples the form of arc described above may be used.

No flux should be employed when fusing rare-metal couples.

Do not allow borax to come in contact with the porcelain lining of a furnace or with porcelain tubes. What borax remains on a base-metal couple may be removed, if desired, by dipping the junction while hot into water. The borax glass will then crack away. In making thermocouple junctions dark glasses should be worn in order that the work may be watched closely. It is also essential that the glasses absorb the ultra-violet light, which may cause severe burns and injure the eyes. Special goggles for this type of work are advertised in the trade journals.

3. USE OF STANDARDIZED COUPLES WITH VARIOUS TYPES OF MEASURING INSTRUMENTS

The temperature-emf relation of a couple may be known and it may be desirable to use the couple with different forms of indicator. Many of the instruments employed are complicated by different compensation devices, the exact operation of which must be understood in order to prevent the introduction of serious error in the observations. In the following we shall assume that the instruments themselves read correctly or that the corrections-to-scale readings are known. If the instruments are graduated in temperature only, it is essential to have a calibration of potential difference at the instrument terminals versus temperature scale. The readings of all galvanometric instruments, except the heat meter (Harrison-Foote method) and the pyrovolter, are affected by changes in line resistance. Hence the resistance of the couple must be equal to that for which the scale of the indicator is designed, or corrections as already described must be applied to the observed deflections. These corrections are assumed

to have been applied when appreciable in the following special cases treated:

(*a*) GALVANOMETER ADJUSTED TO READ ZERO ON OPEN CIRCUIT OR POTENTIOMETER WITHOUT COLD-JUNCTION COMPENSATOR.—If the cold junctions of the couple are at a temperature different from that for which the couple was standardized, a cold-junction correction as discussed above must be added to the observed reading. This also applies to the use of the pyrovolter.

(*b*) GALVANOMETER ADJUSTED TO READ COLD-JUNCTION TEMPERATURE OR EQUIVALENT EMF ON OPEN CIRCUIT.—This adjustment of the open-circuit reading may be either manually or automatically operated, in the latter case usually by a bimetallic spring. The standardization of the instrument itself should be made with the pointer adjusted to the cold-junction temperature employed in the standardization of the couple. If, however, the instrument has been calibrated with a different adjustment, the two calibrations may be coordinated by adding to the emf reading of the instrument calibration or by subtracting from the emfs of the couple calibration the emf on the couple calibration corresponding to the open-circuit temperature reading of the instrument. The latter operation changes the couple calibration to a new basic cold-junction temperature, shown by the open-circuit reading of the indicator.

If the instrument is graduated in emf, it should be adjusted on open circuit to read the emf shown by the couple calibration corresponding to the temperature of the cold junctions at the time of operation. When the instrument is used with several couples, the calibration of all the couples must be referred to the same basic cold-junction temperature.

(*c*) POTENTIOMETER WITH COLD-JUNCTION COMPENSATOR.—The potentiometer compensator adds to the emf developed by the couple an amount equal to the reduction in emf of the couple occasioned by a rise in cold-junction temperature. The magnitude of the compensation may be measured by potentiometers graduated in emf by short-circuiting the emf terminals with a copper wire and then balancing the instrument in the usual manner. The main scale reading gives the amount of the compensation. When the main scale is calibrated with a manually adjusted compensator set to read a given value, the compensator scale is thereby made correct at this reading, though it may be in error at other points. Likewise, an automatic compensator is correct

at the temperature at which it is maintained during a calibration of the instrument. The compensator, if manually operated, as well as the main scale, may be graduated either in emf or temperature. The possible combinations require separate consideration.

(1) *Main Scale and Manually Operated Compensator Graduated in Emf.*—During operation the compensator (corrected for scale error, if necessary) should be set at the emf corresponding to the temperature of the cold junctions at the time of operation, as indicated by the couple calibration regardless of the basic cold-junction temperature employed during the standardization of the couple. If, however, the cold-junction temperature during operation is lower than the basic cold-junction temperature used in the calibration of the couple, the latter should be changed to a lower value, unless the compensator can be set for negative compensation, an adjustment not furnished in the present designs.

(2) *Main Scale and Manually Operated Compensator Graduated in Temperature.*—The instrument should have a temperature-scale-reading-versus-emf calibration, for which the compensator is set to read the temperature of the cold junctions of the couple during its standardization. This makes the compensator reading correct at this setting, although it may have scale errors at other settings. In further use the compensator (corrected for scale error) is set to read the cold-junction temperature. The hot-junction temperature is then obtained by use of the temperature-scale-reading-versus-emf calibration of the instrument and the temperature-emf calibration of the couple.

(3) *Main Scale Graduated in Temperature, Manually Operated Compensator Graduated in Emf.*—The temperature-scale-reading-versus-emf calibration of the instrument is made with the compensator set to read zero. The calibration of the couple is adjusted to the basic cold-junction temperature corresponding to the main-scale reading when the instrument is balanced with the emf terminals short-circuited and the compensator set to read zero. In use the compensator is set at the emf corresponding, on the revised thermocouple calibration, to the operating temperature of the cold junctions, and the hot-junction temperature is obtained by use of the revised temperature-emf calibration of the couple and the temperature-scale-reading-versus-emf calibration of the instrument.

With depressed zero potentiometers—that is, instruments having the lowest main scale reading greater than zero; for example, starting at 500° C or 5 millivolts—it is impossible to obtain a balance

with the emf terminals short-circuited. In this case a temperature-scale-reading-versus-emf calibration of the instrument is made with the compensator set at zero. Select a temperature for which it is most desirable to have a correct scale reading—for example, 1000° C—and note the corresponding emf. Then the calibration of the couple is revised by adding or subtracting from all emf values an amount sufficient to make the above emf value correspond to the chosen temperature. Plot the revised thermocouple calibration and determine the temperature corresponding to zero emf. This is the new basic cold-junction temperature. In the further use of the instrument the compensator is set to read the emf on the revised thermocouple plot corresponding to the operating cold-junction temperature, and the hot-junction temperature is obtained directly from the main temperature scale reading corrected for scale errors. The scale correction is determined by comparing at equivalent emfs the temperature-scale-versus-emf calibration of the instrument with the revised temperature-emf calibration of the couple. Occasionally this method of calibration is desirable for ordinary potentiometers, since it makes a chosen scale reading correct.

(4) *Main Scale Graduated in Emf and Manually Operated Compensator Graduated in Temperature.*—The emf terminals are short-circuited, and with the main scale set to read zero the compensator is adjusted until a balance is obtained. The compensator reading gives the proper basic cold-junction temperature to which the thermocouple calibration is adjusted. Accordingly, for this cold-junction temperature the compensator scale is correct. In further use the compensator dial (corrected for scale error) is set at the operating cold-junction temperature, and the hot-junction temperature is determined from the main scale reading (corrected for scale error) and the revised temperature-emf relation of the thermocouple. Instruments having a depressed zero are used as described in the preceding paragraph.

(5) *Scale Graduated in Emf and Automatic Compensator.*—The compensating emf added to that developed by the couple depends upon the temperature of a nickel resistance which is so located as to acquire the cold-junction temperature of the couple. The calibration of the couple must have a basic cold-junction temperature determined by the temperature of the nickel resistance when the compensating emf is zero, that is, when some chosen scale reading corresponds to the emf at the terminals of the instru-

ment. If inconvenient to determine this by actually varying the temperature of the compensating coil, it may be obtained as follows: Suppose that the chosen scale reading be zero millivolts, this must correspond to zero emf across the instrument terminals. Hence the emf terminals are short-circuited, and a balance is secured, as in an ordinary temperature measurement. The reading e_1 thus obtained shows the amount of compensation for the temperature t_1 of the compensating coil. The calibration data of the thermocouple must be adjusted to a basic cold-junction temperature such that for a hot-junction temperature t_1 the couple develops the emf e_1. This is done by subtracting from all emf values in the original couple calibration the quantity $e_o—e_1$, where e_o is the emf corresponding to the temperature t_1 in the original calibration.

A given compensator can be correct for but one type of couple. To determine the accuracy of its construction, the emf terminals of the instrument are short-circuited and the scale readings for a balance observed with various temperatures of the compensating coil. The relation, scale reading versus temperature of the compensating coil, should be identical to the revised temperature-emf calibration of the couple.

If the instrument is of the depressed-zero type a calibrated potentiometer is connected across the emf terminals and the instrument balanced. The difference between the emfs indicated by the two potentiometers is the same e_1 appearing in the discussion above, and the further procedure is identical. The sign of e_1 is positive if the calibrated potentiometer reads lower than the instrument equipped with the compensator, and negative if higher.

(6) *Scale Graduated in Temperature, and Automatic Compensator.*—A scale-reading-versus-emf calibration must be made. The thermocouple calibration should be adjusted to a basis cold-junc tion temperature equal to that of the compensating coil during the instrument calibration. In further use the temperature of the hot junction is obtained directly from the corrected temperature scale of the instrument. This is determined by comparing the temperature-scale-versus-emf calibration of the instrument at equivalent millivolts with the revised temperature-emf calibration of the couple.

4. STANDARDIZATION OF THERMOELECTRIC INSTRUMENTS

(*a*) LABORATORY STANDARD POTENTIOMETER.—The potentiometer should be calibrated about three months after its manufacture and at intervals of two or three years. Two, or preferably three, standard cells should be available for intercomparison. In case one shows changes relative to the other two, it should be restandardized, and one or more of the cells should be occasionally calibrated, whether or not such variations are detected. The calibration of standard cells and potentiometers should be done either by the Bureau of Standards or the manufacturers. A simple method of intercomparison of cells is as follows: With cell number 1 and the standard cell dial set to the corresponding certified emf, the instrument is balanced against the standard cell in the ordinary manner. Cell number 1 is then replaced by cell number 2, and the *standard cell* dial adjusted until a balance is again obtained. If this reading is not the certified emf of cell number 2, one of the two cells has changed. The cell in error may be detected by an intercomparison with a third cell, unless the three cells have changed similarly, which condition is detected by the periodic standardization of one of the cells. The cells should not be subjected to air currents or other causes of temperature change immediately previous to or during use or intercomparison. They should not be allowed to freeze.

(*b*) ADJUSTMENT OF INSTRUMENTS FOR CALIBRATION.—Instruments having a variable zero or open-circuit adjustment should be set to satisfy the conditions of use, as already described.

(*c*) GALVANOMETERS.—Galvanometers may be calibrated in terms of the potential drop across the instrument terminals, or allowance may be made for a definite external resistance. Fig. 152 shows a convenient wiring diagram. Adjustable resistances R_1 and R_2 are connected in series with a battery B. The galvanometer terminals are connected, with the proper polarity, across R_1. Either one of the resistances is adjusted until the desired scale reading is obtained, and the potential drop across the galvanometer terminals is measured by the standard potentiometer. If the galvanometer is to be calibrated for a definite external resistance, this amount x may be placed in the circuit, as illustrated. If $x = o$ the calibration is in terms of the potential drop across the galvanometer terminals. Observations of instrument reading versus true emf should be made at least at 15 points over the scale, and any range showing irregularities should be further checked.

The resistance R_1 may be varied from 0 to 100 ohms and R_2 from 100 to 10 000 ohms. In general, R_2 should be kept rather large to avoid drawing too much current and to prevent rapid decrease of current due to polarization, if old dry cells are employed. In making the first set up, R_1 should be small (or even zero) and R_2 large. R_1 should never be disconnected from the circuit without first disconnecting the galvanometer or opening the battery circuit at R_2 or B. It should be noted whether the instrument returns to the same open-circuit reading after the calibration, also after the full scale deflection is registered for some time. It is advisable to tap a deflection instrument before each reading during standardization in order to avoid sticking of the coil in its

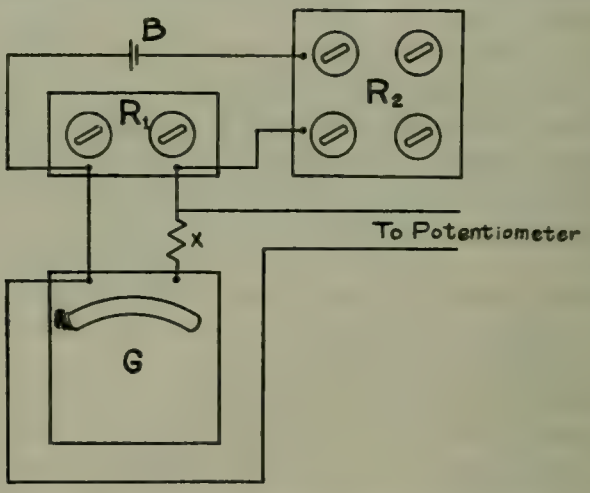


FIG. 152.—*Wiring diagram for standardization of galvanometric indicators*

mounting. Instruments equipped with a bimetallic spring zero adjuster should be calibrated at constant room temperature.

The temperature coefficient of a galvanometer may be observed by calibrating twice at two widely different room temperatures. Sufficient time—several hours— must be allowed for the instrument to reach a uniform temperature.

A bimetallic spring cold-junction compensator may be tested by varying the temperature of the instrument and observing the corresponding scale readings on open circuit. The relation, emf scale reading versus temperature, so obtained, must be identical to the temperature-emf relation of the couple based on the cold-junction temperature t_o where t_o is the temperature of the galvanometer for a zero-emf scale reading. If the scale is graduated

in degrees, the pointer, on open circuit, should indicate the temperature of the galvanometer.

(*d*) PORTABLE AND RECORDING POTENTIOMETERS.—The emf or thermocouple terminals of the instrument are connected to the emf terminals of the laboratory standard potentiometer, + to + and − to −. Both instruments are balanced as usual against their individual standard cells, and with various settings of the scale of the instrument under test the true emf is obtained from the reading of the standard potentiometer. Different portable potentiometers may be similarly intercompared, in which case it is desirable to short-circuit one of the galvanometers after balancing against the standard cell in order to increase the sensitivity.

(*e*) POTENTIOMETER WITH COMPENSATOR GRADUATED IN TEMPERATURE.—The compensator is set at the desired basic cold-junction temperature of the couple, and the main scale is tested as above. This makes the compensator scale correct at this particular setting. To calibrate the compensator scale, the main scale is set at any definite position, preferably greater than the highest setting on the compensator scale, and several measurements are made of the emf for different compensator settings, one setting being the same as that used for the main scale calibration. Each reading is subtracted from the value determined by this latter setting. The compensator readings versus the emf differences should be equivalent to the temperature-emf relation of the couple. If these do not agree sufficiently well, the compensator scale should be standardized in terms of scale reading versus true cold-junction temperature, and the necessary correction applied in subsequent use each time the compensator is set.

(*f*) POTENTIOMETER WITH COMPENSATOR GRADUATED IN EMF.—The main scale is calibrated with the compensator set at zero. This makes the zero setting of the compensator correct. To calibrate the compensator scale, the main scale is set at any definite position, preferably greater than the highest setting on the compensator scale, and measurements are made of the emf for various compensator settings. Each reading is subtracted from the value observed with the compensator set at zero. This gives the calibration of the scale reading versus true emf for the compensator.

(*g*) POTENTIOMETER WITH AUTOMATIC COMPENSATOR.—These are calibrated in the same manner described for simple potentiometers, the temperature of the compensating coil being noted and

maintained constant during the observations.[48] If the scale is graduated in emf and accurately made, true emf across the potentiometer terminals is indicated for some definite temperature of the compensator, but at other temperatures this is not the case. Hence it is desirable to know the basic temperature of the compensator for which the scale graduation was made. This may be determined for an instrument which does not have a depressed zero by short-circuiting the emf terminals and varying the temperature of the compensator coil until a balance is obtained with an emf scale reading of zero. The temperature-emf relation of the couple must be adjusted for this basic cold-junction temperature.

It is not always convenient to vary the temperature of the compensator. The instrument may be tested with the temperature of the compensator that of the room, preferably at the approximate mean cold-junction temperature to be employed in subsequent use. A calibration of emf scale readings versus true emf is made in the manner described above, and the temperature-emf relation of the couple is adjusted to the basic cold-junction temperature equivalent to that of the room during the standardization of the potentiometer. By comparing the temperature-emf relation of the couple to the scale-reading-versus-true-emf relation for the instrument, a final table may be prepared of scale reading versus temperature. The general procedure is identical when the potentiometer scale is graduated in temperature.

(*h*) INSTRUMENTS EMPLOYING THE HARRISON-FOOTE METHOD.—If the laboratory potentiometer is of the low-resistance type, such as the Leeds & Northrup precision thermocouple potentiometer, the "heat meter" is most easily and accurately standardized by connecting the emf terminals of the two instruments together, + to + and − to −. After balancing the potentiometer against the standard cell its galvanometer terminals are short-circuited and the potentiometer key is closed. The two instruments will then read approximately the same. The potentiometer is set to give the desired deflection of the heat meter, and before each reading the heat meter must be adjusted by the usual method to compensate for external resistance. The potentiometer setting gives the true emf corresponding to the scale reading of the heat meter. Heat meters having an adjustable resistance of 15 ohms may be calibrated in the above manner by means of the Leeds &

[48] The accuracy of the compensator may be determined by the method given on page 249.

Northrup type K potentiometer, provided the ratio coils are so set that the fourth dial step is not exceeded.

If a potentiometer of high resistance must be used, the wiring diagram shown in Fig. 152 may be employed, in which x is zero and R_1 is less than 15 ohms (the value of r_5 in Fig. 21*a*). The resistances R_1 and R_2 are adjusted for the desired deflection, the heat meter being set in the usual manner to compensate for external resistance. The true emf corresponding to the observed scale deflection is obtained by measuring the potential drop across R_1 with the potentiometer, *while the heat meter is disconnected* from the circuit. A good dry battery or storage cell is necessary for these determinations. If R_1 is less than 0.1 ohm and the potentiometer is connected directly across R_1, it is unnecessary to disconnect the heat meter.

A less precise method of standardization is to set the adjustable resistance of the heat meter to the marked position and follow the procedure described for calibrating a galvanometer in terms of the potential drop across its terminals.

5. CALIBRATION OF COUPLE AND INDICATOR AS A UNIT

Often it is desirable to calibrate a couple and indicator as a unit, the calibration giving the relation between scale reading and true temperature.

(*a*) GALVANOMETRIC INSTRUMENTS.—The calibration automatically takes account of line resistance. In future use, however, it is important that the resistance of the circuit does not change, especially if the instrument is of low resistance. If the galvanometer is always adjusted to read zero on open circuit, the cold-junction temperature of the couple during calibration is the basic cold-junction temperature to which subsequent cold-junction temperatures must be corrected, as described on page 246. If the galvanometer is graduated in temperature in the low range, the pointer may be adjusted to read the cold-junction temperature both during calibration and subsequent use. This avoids the necessity for applying cold-junction corrections. Galvanometers equipped with a bimetallic spring compensation operate the zero shift automatically, and should be so adjusted that on open circuit the instruments indicate the cold-junction temperature.

(*b*) POTENTIOMETERS.—When a simple potentiometer without compensator is calibrated with a couple, as a unit, the cold-junction temperature during calibration is the basic cold-junction tem-

perature of the couple to which subsequent cold-junction temperatures must be corrected, as described on page 246. When the potentiometer has a compensator graduated in temperature, the compensator is set to read the cold-junction temperature of the couple both during calibration and subsequent use. When the main scale of the potentiometer and the compensator are both graduated in emf, a setting of the compensator should be so chosen during calibration that in subsequent use any likely cold-junction temperature may be compensated for. Usually the cold-junction temperature is not less than o° C. Hence one method is to set the compensator at zero and calibrate with a cold-junction temperature of o° C. If the calibration must be made with the cold-junction temperature that of the room, it is not desirable to set the compensator at zero, because in subsequent use the room temperature may decrease, and no compensation could be afforded, since negative compensation is not provided for. Hence during calibration the compensator should be adjusted to such a position that it will read zero when the cold-junction temperature is o° C or the lowest temperature, t_0, likely to be encountered. This position may be estimated from a knowledge of the temperature-emf relation of the general type of couple employed. In future use of the pyrometer the compensation for cold-junction temperature must be referred to the basic cold-junction temperature for which the compensator reads zero; that is, o° C or t_0 in the above example. If the main scale is graduated in temperature, short-circuit the emf terminals, set the main scale to read the cold-junction temperature to be used in the calibration, and balance by adjusting the compensator. This gives the proper compensator setting if the main temperature scale is approximately correct for the type of couple employed. In subsequent use the compensator may be set by the same method, or the compensation may be estimated from a knowledge of the temperature emf relation of the general type of couple used. This relation must be referred to the basic cold-junction temperature determined by the reading of the main scale when the instrument is balanced with the emf terminals short-circuited and the compensator set at zero.

6. STANDARDIZATION OF OPTICAL PYROMETERS

An optical pyrometer may be standardized by either a primary or a secondary method. A precise and satisfactory primary calibration requires considerable technique, but the secondary

method is sufficient except for special research work in the laboratory, when the highest possible precision is desired. In the secondary method the pyrometer under test is compared with an instrument which has been accurately standardized. The primary standard optical pyrometer of this Bureau, shown in Fig. 153, operates on the disappearing-filament principle, since this method is capable of the highest precision. In the following sections are considered methods of secondary standardization, after which a brief statement of the method employed for primary standardization is presented.

(*a*) SECONDARY STANDARDIZATION.—(1) *Comparison Source.*—A constant and uniform source of light is required upon which the two pyrometers are sighted. It is not essential that this

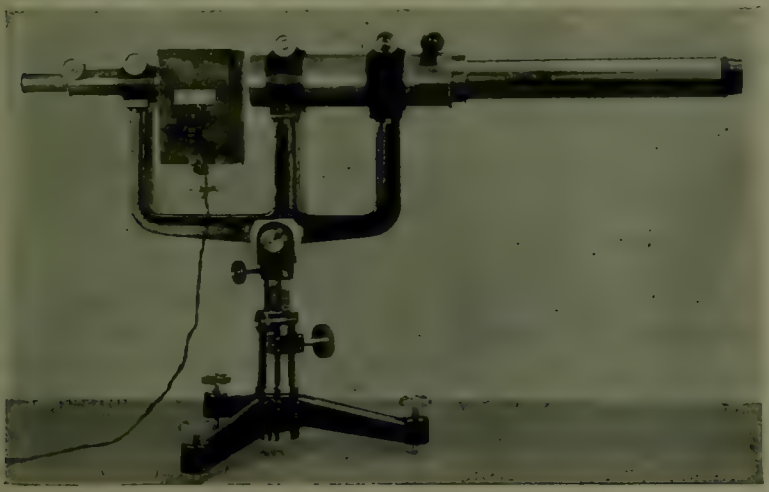


FIG. 153.—*Bureau of Standards precision optical pyrometer*

source be a black body, but if the radiation is very selective only pyrometers of the same type or instruments using the same wave length should be compared. Fig. 154 illustrates a black-body furnace suitable for temperatures up to 1550° C. Its construction is similar to that described under Fig. 134. A series of diaphragms of successively smaller openings lead into the interior inclosure, from which the radiation is emitted. The pyrometer is focused on the diaphragm *A*. The angle *bAc* is properly chosen somewhat greater than the cone of rays required by the pyrometer; otherwise one of the front openings may act as a diaphragm to the optical system and cut down the aperture, thus giving an erroneous calibration. For temperatures up to 2500° C the Arsem furnace,

already described, is useful. Thus, in Fig. 139 the sample *A* may be removed or both *A* and *B* may be replaced by a graphite crucible. A small furnace for operation up to 1600° C may be made by winding the inside walls of an alundum crucible (front, back, and side walls) 3.5 by 3.5 cm, with 0.6 mm platinum or platinum-rhodium wire. The turns of wire are held in place by a thin layer of alundum cement, and the radiation is emitted from a front opening about 1 cm in diameter. If no insulation is employed, the temperature of the furnace may be readily adjusted to any desired value, and by using battery current can be maintained constant.

A carbon-strip lamp, consisting of a thin carbon ribbon about 8 mm wide, mounted in an evacuated glass bulb, may be operated up to 1800° C, and the radiation is sufficiently black for ordinary calibration work, even with pyrometers using different quality of red glass. The General Electric Co. has made lamps of this character, requiring from 10 to 15 amperes for 1800° C. The temperature distribution across the strip is never very good, and great care is required that both pyrometers are sighted on exactly the same portion of the ribbon.

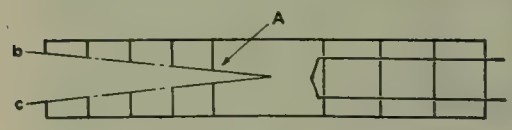


FIG. 154.—*Black-body furnace*

A strip of pure sheet nickel electrically heated in air forms a tough coating of oxide which does not scale. This type of source possesses the advantage that it may be made as large and uniform as required, but it is unsatisfactory for operation above 1200° C. The Nela laboratory has developed the tungsten-strip lamp, which may be operated up to 2500° C. A ribbon of tungsten about 2 mm wide and 4 cm long is mounted in a gas-filled bulb. While the source is not black, its emissivity at various wave lengths is well known and may be corrected for when required. The width of the source, however, is so small that its use is practically confined to the disappearing-filament pyrometer, and if the instruments compared have the same type of red glass, as is usually the case, the departure of the source from black-body conditions is of no importance.

(2) *Disappearing-Filament Pyrometer.*—Fig. 155 illustrates the arrangement for comparing the pyrometer on the left with a standard instrument on the right. The comparison source is the small furnace described above, or a carbon or tungsten strip lamp. Since the radiation intensity from such a strip varies with the angle

ϑ at which the pyrometer is sighted, it is desirable to make both ϑ and ϑ' as small as convenient and as nearly equal as possible. It is also necessary to focus both pyrometers on the same portion of the strip. This may be done by locating some peculiarity of the strip visible in each pyrometer, or the pyrometer lamps may be heated to about 1300 or 1400° C and the images of the filaments superposed on the strip at the point desired. If the furnace shown in Fig. 154 is employed, it is inconvenient to sight both pyrometers at the same time. A convenient means may be devised for shifting either the furnace or the pyrometers by the proper

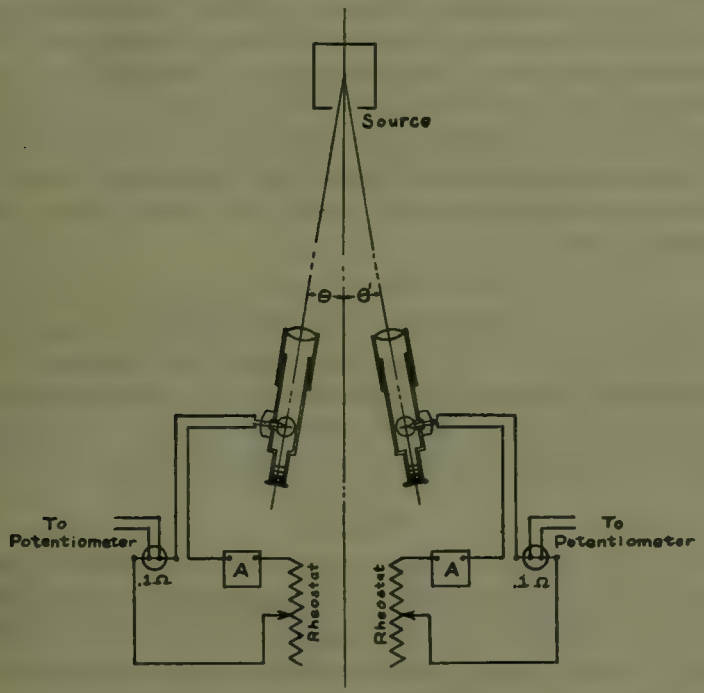


FIG. 155.—*Comparison method of standardizing optical pyrometers*

amount, so that readings may be made first with one instrument and then with the other. With the arrangement illustrated two observers may take readings simultaneously, thus eliminating any effect due to change in temperature of the source. If a new pyrometer lamp is employed, it should be first annealed at not less than 2000° C for 20 hours for a tungsten filament and 1800° C for a carbon filament. Tungsten filaments with welded terminals are now almost exclusively used.

For an approximate calibration the current may be measured by means of an ammeter. but for a precision calibration it is

desirable to use a potentiometer. Resistance standards of 0.1 ohm are connected in series with the lamp, and the potential leads are carried to a potentiometer. The currents usually required are from 0.2 to 0.6 ampere, which, over a 0.1 ohm, give a potential drop of from 20 to 60 millivolts, a convenient range for the laboratory thermocouple potentiometer. The use of a deflection potentiometer for the current measurements is very satisfactory, this instrument furnishing sufficient accuracy (0.1 per cent) and possessing nearly the convenience of an ammeter.

The current measurements with the standard instrument are converted to temperature from the primary calibration chart, whence a plot may be made of current versus temperature for the instrument under test. The plot may be prepared by drawing the best smooth curve through the observed data. It is, however, desirable in precise work to use a definite form of curve for which the constants are computed from the experimental data. The following equation holds very satisfactorily from 700 to 1500° C for carbon lamps:

$$i = a + bt + ct^2$$

where the constants a, b, and c are determinable by three standardization points. This equation applied to tungsten lamps should not be extrapolated below the lowest observed point, nor for more than 100° C above the highest point. An equation slightly more satisfactory for tungsten has the form:

$$i = at + be^{ct}.$$

This equation may be extrapolated 100° C in the low range, but errors of 5° C may result from an extrapolation of 100° C in the high range. Accordingly, for precision work it is desirable to employ the more difficult cubic equation with four constants, viz.:

$$i = a + bt + ct^2 + dt^3.$$

The constants are determinable by four standardization points, but it is usually desirable to obtain at least 10 comparison points from which the best curve may be computed by least squares in the following manner: The values of i, t, t^2, t^3, t^4, t^5, t^6, it, it^2, it^3, are tabulated for each calibration point and the sums of all the n values of i are denoted by Σi, of all the n values of t by Σt, etc. These sums are substituted in the following normal equations, which are solved for a, b, c, and d: Normal equations:

$$
\begin{aligned}
an &+ b\Sigma t &+ c\Sigma t^2 &+ d\Sigma t^3 = \Sigma i \\
a\Sigma t &+ b\Sigma t^2 &+ c\Sigma t^3 &+ d\Sigma t^4 = \Sigma it \\
a\Sigma t^2 &+ b\Sigma t^3 &+ c\Sigma t^4 &+ d\Sigma t^5 = \Sigma it^2 \\
a\Sigma t^3 &+ b\Sigma t^4 &+ c\Sigma t^5 &+ d\Sigma t^6 = \Sigma it^3
\end{aligned}
$$

Thus, knowing a, b, c, and d, the equation of the lamp may be computed for various temperatures, and a curve may be drawn through these points, or a table of current versus temperature may be calculated.

For extending the range of the pyrometer above 1400° C absorption glasses calibrated as described on page 106 are employed. In ordinary work the value of A so obtained is a constant, but in precision testing it is found that A usually varies somewhat with the temperature of the source sighted upon, as will be briefly considered later.

If the pyrometers are calibrated separate from the ammeters, the ammeters should be also tested at several points. This is readily done by connecting a 0.1-ohm resistance standard in series with the ammeter and measuring the potential drop across this resistance for various settings of the ammeter, whence a table of corrections may be prepared for the instrument. This method is more desirable than to calibrate the ammeter and pyrometer as a unit, since if changes in the calibration occur it is then readily apparent whether the lamp or the ammeter is at fault.

In the comparison method of calibration of optical pyrometers no corrections need be applied for absorption of light by glass windows, glass bulbs of strip lamps, etc., since both instruments are affected similarly.

It is occasionally desirable to check an instrument at a single point. Any convenient source of light is satisfactory for this purpose. For example, a stone block in a neighboring building, illuminated by sunlight, or a concrete sidewalk, etc., furnishes a source of apparent temperature from 700 to 1200° C. Such sources however, are far from black, and it is very important that the same type of red glass be used in each pyrometer so compared.

(3) *Wanner Pyrometer.*—This instrument requires a much larger source than the disappearing-filament pyrometer, and hence neither a carbon nor tungsten strip lamp is satisfactory. A wide nickel strip may be used, but for a sufficient temperature range either the black-body furnace or the Arsem furnace is necessary. Metal strips are also objectionable for use with the Wanner, since the emitted light is polarized and readings will be affected by the

orientation of the instrument. The source sighted upon must be uniformly heated, and the instrument should be carefully aligned, so that the standard pyrometer may be focused on the center of the field used by the Wanner.

The relation between the log tan of the angular reading and the reciprocal of the absolute temperature is linear. The brightness of the black-body source sighted upon is matched against that of the electric lamp which burns at a constant brightness X. Since the photometric fields illuminated by the lamp and furnace are polarized at right angles, we obtain the following equation for the condition of a match, where J is the intensity of the source at the absolute temperature ϑ, and φ is the angle through which the analyzer or eyepiece is rotated.

$$J = X \tan^2 \varphi$$

At the normal point some definite angle φ_0 corresponds to an intensity J_0 thus:

$$J_0 = X \tan^2 \varphi_o,$$

Hence

$$J = J_o \frac{\tan^2 \varphi}{\tan^2 \varphi_o},$$

or $$c_1 \lambda^{-5} e^{\frac{-c_2}{\lambda \vartheta}} = c_1 \lambda^{-5} e^{\frac{-c_2}{\lambda \vartheta_0}} \frac{\tan^2 \varphi}{\tan^2 \varphi_0}.$$

Accordingly $\log \tan \varphi = \left[\frac{c_2 \log e}{2 \lambda \vartheta_o} + \log \tan \varphi_0 \right] - \frac{c_2 \log e}{2 \lambda \vartheta}$,

or $\log \tan \varphi = a - \frac{b}{\vartheta}$ where a and b are positive constants determinable by two standardization points, although more points are always desirable.

The adjustment for the normal point is made as described on page 100, and the instrument is then compared with the standard at about 10 different temperatures well distributed over the angular scale of the pyrometer. After this comparison the normal setting is again determined to assure that no change has taken place. The reciprocal of the absolute temperature $\frac{1}{\vartheta}$ is plotted against the logarithm of the tangent of the angle, log tan φ, as shown in Fig. 156, and the best straight line is drawn through the observed points. The temperatures corresponding to the various angles are then read from this plot, and a table of temperature versus angle from $\varphi = 10°$ to $\varphi = 80°$ is prepared for subsequent use.

If the instrument has a temperature scale engraved on the dial, it is unnecessary (although still preferable) to make the above computations. The correct temperature may be plotted against the temperatures indicated by the Wanner and a correction chart prepared similar to those furnished with precision voltmeters. However, since the curve φ vs. ϑ is not linear, but

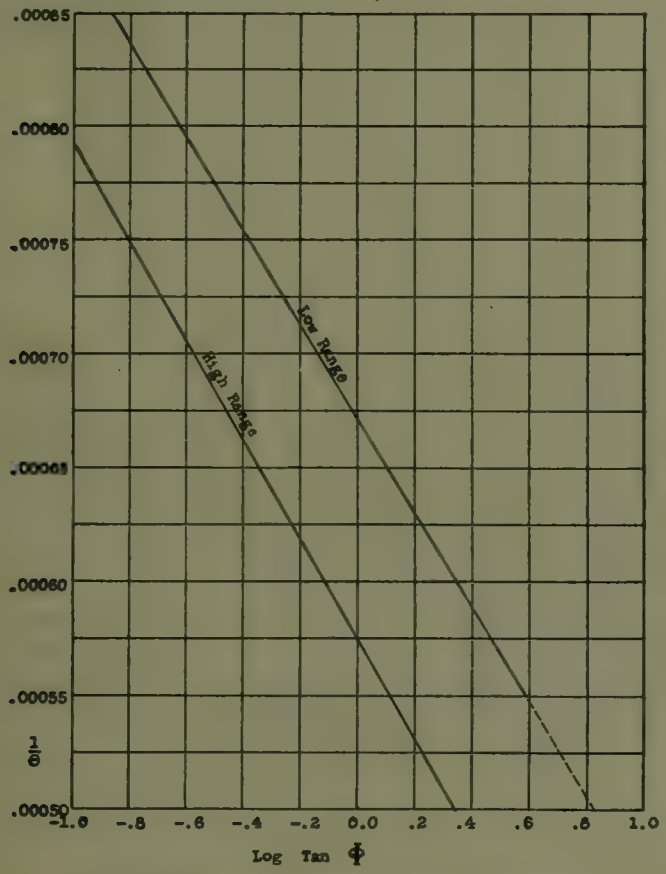


FIG. 156.—*Calibration plot for Wanner pyrometer*

S-shaped, it is difficult to interpolate corrections on the temperature scale directly unless a large number of standardization temperatures are selected.

The high-range Wanner is equipped with an absorption glass which may be standardized in the manner described for the absorption glass of the disappearing-filament pyrometer, page 106, or the instrument may be standardized directly with the absorption glass.

The Wanner pyrometer contains several set screws, the movement of any one of which may throw the instrument out of adjustment. In general, it is inadvisable to attempt to readjust the optical parts, as the process is difficult and complicated. If the dividing line in the field is poor or if it is impossible to obtain a uniformly illuminated field, it is likely that the Wollaston prism has been moved or requires recementing with Canada balsam. Occasionally the set screw on the analyzer loosens, but this adjustment is easily made in the laboratory. Sight on any constant source and match the two fields by setting the analyzer in the first quadrant, as is done in an ordinary temperature measurement. Then make a setting with the pointer in the second quadrant. This can be done after removing a stop screw either on the dial or on the bearing upon which the eyepiece turns. The sum of the two angular readings should be 180°, and the analyzer set screw is adjusted until this is the case.

(4) *Fery Optical and F and F Pyrometer*..—These instruments employ a linear wedge of black glass which is moved across the photometric field until a match is secured. With the Fery instrument the motion of the wedge is read from a linear scale, and with the *F* and *F* this motion is transferred by a gear to a circular scale on the dial plate. In either case, if the scale is divided into equal spaces—for example, circular degrees for the *F* and *F*—the scale reading plotted against the reciprocal of the absolute temperature is approximately a straight line. Thus, referring to Fig. 58, the source *F* is properly adjusted in the manner described to a constant brightness *X*. If the instrument is sighted on a black body of temperature ϑ, we obtain for the condition of a photometric match:

$$Tc_1\lambda^{-5}e^{-\frac{c_2}{\vartheta\lambda}}=X$$

where *T* is the transmission coefficient of the wedge. If strictly monochromatic light were employed (the condition for the narrow band of radiation actually used is not seriously different as far as practical considerations are concerned), the transmission coefficient of the wedge bears the following relation to the thickness x and to the angle α, which is proportional to this thickness, *K* and *K'* being constants:

$$T=e^{-Kx}=e^{-K'\alpha}\ .$$

On substituting this value of T in the above equation and taking the logarithm of each member we obtain:

$$\alpha + \left[\frac{\log_e X}{K'} - \frac{\log_e c_1 \lambda^{-5}}{K'}\right] = -\frac{c_2}{K'\lambda}\frac{1}{\vartheta}$$

or $\alpha - P = -Q/\vartheta$ where P and Q are positive constants. The constant X is fixed by the normal setting of the pyrometer. Thus some particular angle α_0 is made to correspond to a temperature ϑ_0; whence

$$e^{-K'\alpha_0} c_1 \lambda^{-5} e^{-\frac{c_2}{\lambda\vartheta_0}} = X = e^{-K'\alpha} c_1 \lambda^{-5} e^{-\frac{c_2}{\lambda\vartheta}}$$

or

$$\alpha - \left[\alpha_0 + \frac{c_2}{K'\lambda\vartheta_0}\right] = -\frac{c_2}{K'\lambda}\frac{1}{\vartheta}$$

thus, $\alpha - P = -Q/\vartheta$, as before.

Hence the relation between the angle α and the reciprocal of the absolute temperature $1/\vartheta$ is linear. The adjustment on the normal point is made in the manner already described, and the instrument is compared with the standard pyrometer at a series of temperatures. The angle α is plotted against $1/\vartheta$, and the best straight line drawn, as shown in Fig. 157; whence a table or plot of α versus $t°$ C may be prepared. If the instrument is graduated in temperature a simple table of corrections may be determined without plotting the data in terms of angular readings.

(*b*) Primary Standardization.—A secondary calibration of a properly designed disappearing-filament pyrometer may be easily made with an accuracy of 5° C and, if necessary, 2° C, or even better. Hence there is no reason for making a primary calibration unless the highest possible precision for research work is required.

A primary calibration requires the use of a black body, and every precaution must be taken to assure that the radiation is as nearly black as possible. Two conditions should be fulfilled; the opening into the hollow inclosure, in general, should be small compared to the size of the inclosure, and the walls of the inclosure must possess a uniform temperature. The black body shown in Fig. 158 satisfies these conditions. The crucible and radiator are constructed of graphite, and the radiation from the inclosure B is emitted through the opening C. The space D is filled with metal, such as copper. The bath of molten metal of high thermal conductivity insures temperature uniformity over the entire radiator. The radiation is so black that all detail of contour

vanishes, and, in order to distinguish the opening *C* for focusing and adjusting the pyrometer, it is necessary to allow a drop of water to fall into *B*, when momentarily *C* appears in the field as a dark spot. This form of crucible has been made in smaller sizes for use in the Arsem furnace, but it has been found that no appreciable sacrifice in blackness is offered by the much-easier constructed crucible shown in Fig. 144. Using this type of crucible a primary calibration is made by observing, as described on page 211,

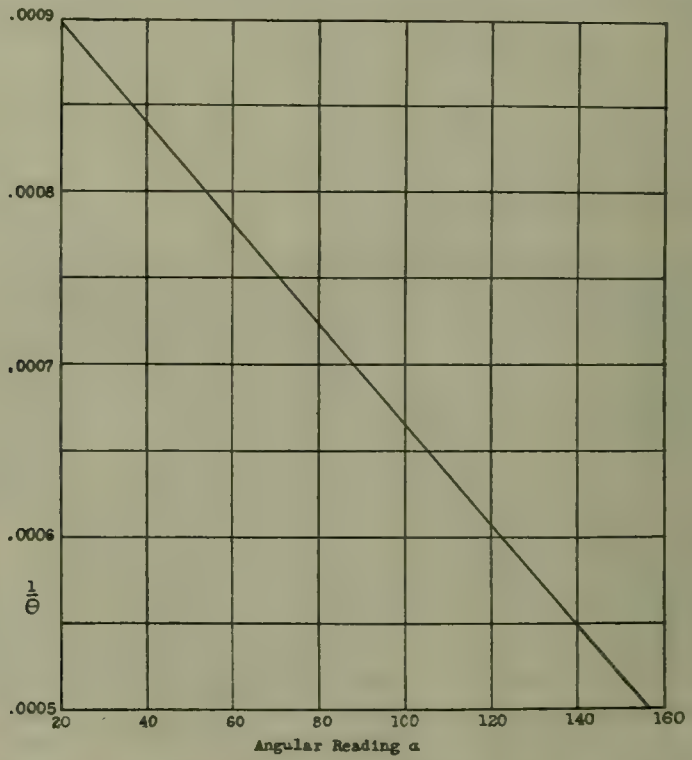


FIG. 157.—*Calibration plot for F and F or Fery optical pyrometer employing a linear wedge*

the freezing or melting curves of several metals the melting points of which are accurately known. The metals chosen are aluminum [49] (99.66 per cent pure) 658.7° C; copper-silver eutectic, 779° C; silver, 960.5° C; and copper, 1083° C, or gold, 1063° C. The time of melting or freezing is adjusted to about 20 minutes, and at least 30 or 40 observations are made during the change in phase. If a glass window is used on the furnace it is necessary to apply small corrections to the above melting points to allow for

[49] See B. S Circular No. 66.

the absorption of the glass, as described on page 117. The experiments are repeated several times, and the mean values of the currents determined potentiometrically are substituted in the cubic equation $i = a + bt + ct^2 + dt^3$, which is solved for the constants a, b, c, and d. This calibration curve is interpolated and extrapolated from 650 to 1400° C. The data so obtained are carefully compared, especially above 1200° C, by sighting into the Lummer Kurlbaum black body shown in Fig. 154, the temperature of which is measured by two accurately standardized rare-metal thermocouples. If any differences exist, the latter observations are given weight and a new calibration curve is recomputed. However, if the diaphragms have progressively smaller openings into the interior of the furnace, so that good black-body conditions obtain, the two methods of standardization are in excellent agreement.

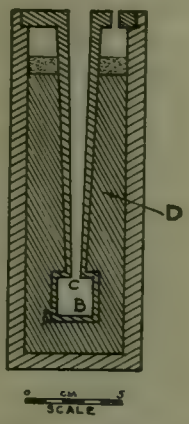


FIG. 158.—*Black body immersed in molten metal*

The precision calibration of the absorption device for extending the range of the pyrometer above 1400° C is a complicated and laborious process. The quantity $A = 1/\vartheta - 1/S$, discussed on page 106, is not, in general, a constant, but, with increasing temperature of the source, numerically increases for the usual type of absorption glass and decreases for a sectored disk. This is due to the fact that strictly monochromatic light is not employed. The red glass ocular transmits a band of light, as illustrated by Fig. 159.

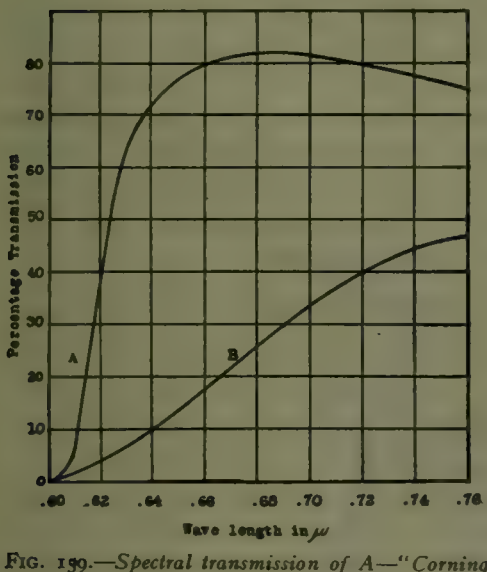


FIG. 159.—*Spectral transmission of A—"Corning High-Transmission Red" marked 150 per cent, thickness 5 mm; B—Jena red glass No. 2745, thickness 3.2 mm*

As the temperature of the source sighted upon through such a glass increases, the color of the glass changes slightly from a deep red to a bright red, and this change in effective wave

length of transmission must be considered. It is necessary to introduce several definitions before the calibration of absorption devices can be discussed. Visibility, $V = f(\lambda)$, is the relative sensitivity of the normal eye to equal intensities of radiation of different wave lengths. Luminosity, $L = f(\vartheta)$, is the luminous intensity of a heterochromatic source, and is proportional to the area under the curve obtained by plotting the product of visibility and energy intensity against the wave length.[50] The effective wave length λ_L of a color filter or screen is that wave length for which a small change in temperature of the black-body source sighted upon produces the same fractional change in luminosity and in the energy intensity at this wave length, as viewed and measured through the screen. This definition is expressed mathematically as follows:

$$\text{Effective wave length} = \lambda_L = \frac{\int_0^\infty T_\lambda V_\lambda J_\lambda d\lambda}{\int_0^\infty \frac{T_\lambda V_\lambda J_\lambda}{\lambda} d\lambda} \qquad (1)$$

where T_λ is the transmission coefficient of the color screen and J_λ is the energy intensity of the black-body source at the wave length λ. Thus λ_L is the λ – coordinate of the center of gravity of the area under the curve obtained by plotting $T_\lambda V_\lambda J_\lambda / \lambda$ versus λ. This may be computed for a series of temperatures, for any particular red glass, by graphical means, as described elsewhere.[51] As an example of the variation of λ_L with ϑ, the following table for Jena red glass No. 2745, thickness 3.2 mm, is illustrative. The variation is much less with the Corning red glass.

Effective Wave Length of a Red Glass Screen

ϑ	λ_L
° absolute	μ
1000	0.6607
1200	.6560
1400	.6532
1600	.6513
1800	.6498
2000	.6487

[50] If luminosity in watts cm^2 is divided by the mechanical equivalent of light, the brightness of the source in candles /cm^2 is obtained.

[51] Foote, B. S. Sci. Papers No. 260.

Analogous to the simple Wien equation strictly applicable only for monochromatic light, the following equation may be written as a definition of the *mean effective wave length* λ'_L when an absorption device is employed with the pyrometer using heterochromatic radiation:

$$\frac{1}{\vartheta} - \frac{1}{S} = \frac{\lambda'_L \log_{10} Tr}{c_2 \log e} = A(\vartheta) \tag{2}$$

In this equation ϑ is the true absolute temperature of the blackbody source, and S is the absolute temperature measured through the absorption device of effective transmission Tr. The effective transmission of the absorption device having a transmission coefficient $T_\lambda' = f(\lambda)$ is defined as the ratio of the luminosity of the source viewed through the red glass to the luminosity of the source viewed through the absorption device and the red glass, as follows:

$$Tr = \frac{\int_0^\infty T T' V J(\lambda, \vartheta)\, d\lambda}{\int_0^\infty T V J(\lambda, \vartheta)\, d\lambda} = \frac{\int_0^\infty T V J(\lambda, S)\, d\lambda}{\int_0^\infty T V J(\lambda, \vartheta)\, d\lambda}. \tag{3}$$

A definite relation holds between λ'_L and λ_L expressed by equation (4)

$$\frac{1}{\lambda'_L} = \frac{\int_{1/S}^{1/\vartheta} \frac{d\left(\frac{1}{\vartheta}\right)}{\lambda_L}}{\frac{1}{\vartheta} - \frac{1}{S}} \tag{4}$$

Whence the reciprocal of the mean effective wave length is equal to the average value of the reciprocal of the effective wave length for the system (red glass + black body) between the limits $1/S$ and $1/\vartheta$. For all red glasses actually used this mean is equivalent within 1 part in 6000 to 10 000 to the simple mean of the effective wave lengths at two temperatures ϑ and S.

The method of applying the above equations to the use of a sectored disk is briefly as follows: Suppose the transmission coefficient of the disk as determined by a dividing engine is 0.01. Hence from equation (3) $Tr = T' = 0.01$. We shall compute the value of A corresponding to the true temperature 2000° abs. The effective wave length of the special Jena red glass described, for $\vartheta = 2000°$, is 0.6487 μ. This is not the value of λ'_L, which is a mean of λ_L at S and λ_L at ϑ. However, as a first approximation

let $\lambda'_L = 0.6487$. On substituting equivalent values in equation (2) we obtain:

$$A = \frac{1}{2000} - \frac{1}{S} = \frac{0.6487 \log 0.01}{(14350)(.43429)}$$

or $S = 1412°$ abs. for a first approximation.

By interpolation of the data in the above table we find that for a temperature of 1412° the effective wave length is $\lambda_L = 0.6531\mu$. The mean of λ_L for 1412° and λ_L for 2000° is $\lambda'_L = 0.6509\mu$. Substituting this value in equation (2):

$$A = \frac{1}{2000} - \frac{1}{S} = \frac{0.6509 \log 0.01}{(14350)(.43429)} = -0.0002089$$

or $S = 1411°$ abs. for a second approximation.

On carrying through higher approximations the value of S (hence λ'_L) remains unchanged; thus $A_{2000} = -0.0002089$ as above. The quantity A may be computed in this manner for various temperatures of the source, giving the following illustrative table:

Variation of A with Temperature of Source

ϑ	A
° abs.	
1400	−0.0002105
1600	− .0002098
1800	− .0002093
2000	− .0002089

The calibration of an absorption glass is more difficult, because not only does the mean effective wave length change with the temperature but also the effective transmission Tr of equations (2) and (3). The value of Tr as a function of ϑ may be determined photometrically, or the integrals in equation (3) may be evaluated graphically and a curve drawn of log Tr versus ϑ or S. However, the accuracy of such determinations is not sufficient. It is more desirable to measure directly ϑ and S up to as high a value of ϑ as is possible without danger of altering the lamp calibration—that is, $\vartheta = 1700$ or 1800° abs.—and to extrapolate the observed curve of $A = 1/\vartheta - 1/S$ versus ϑ, using the same form of curve as that obtained by the graphical computation of $\lambda'_L \log Tr$. Check observations at the higher extrapolated temperatures may be made by comparisons with a calibrated sectored disk.

Foote, Mohler, and Fairchild [52] have showed that it is possible to obtain an absorption glass for which A is a real constant. Such

[52] J. Wash. Acad. Sci., 7, p. 545; 1917.

a glass must have a transmission coefficient of the form $T'_\lambda = e^{K/\lambda}$ where $K/c_2 = A$.

Absorption glasses are very convenient for precision determinations, but their calibration must be made with exceedingly great care. Moreover, the transmission coefficient of an absorption glass depends to some extent upon the room temperature, thus necessitating further corrections not here considered. Many of these difficulties are avoided by using the more inconvenient sectored disk, but this method is also subject to uncertainties, as, for example, the proper location of the sector in the line of sight when a small source is sighted upon. It is accordingly desirable in precision work, such as the establishing of the high-temperature scale, to employ both methods.

There are other means of standardizing an optical pyrometer. For example, a black body may be maintained at the melting point of palladium and a calibration effected by the use of several sectored disks of measured aperture. A more detailed discussion, however, of this field of pyrometry and of the effective wave length entering into precision determinations of emissivity of nonblack radiators is beyond the scope of the present treatment.[53]

7. STANDARDIZATION OF RADIATION PYROMETERS

For precision work the radiation pyrometer must be standardized and used with extreme care. It has been found [54] that many of these instruments are subject to peculiar errors requiring for their elimination rigid specifications in the method of calibration and use.

When a radiation pyrometer is exposed to the radiation from a source at a constant temperature, the instrument does not indicate immediately the temperature of the source, but exhibits a certain time lag during which the receiving system is heating up. Instruments are constructed in which to all practical purposes the equilibrium position is attained in a few seconds, but occasionally one finds receivers for which this is not the case. The readings may continue to increase for 10 to 30 minutes, and sometimes increase for a few moments and then decrease appreciably. These effects should be investigated, and, if important,

[53] Worthing, Trans. A. I. M. M. E.; 1919. Forsythe, Trans. A. I. M. M. E.; 1919. Foote, Fairchild, and Mohler, Absorption Glasses, B. S. Sci. Papers (in preparation).

[54] Burgess and Foote, B. S. Sci. Papers No. 250.

the instrument must be calibrated and used with a definite time of exposure.

The most serious error to which radiation pyrometers are subject is that arising from stray radiation. Some of the heat rays from the source are reflected down the walls of the telescope tube, eventually reaching the receiver. The walls near the receiver become warm and reradiate to the couple. The result of such stray radiation is to increase the readings as the size of the source is increased or the sighting distance decreased. Thus Burgess and Foote found for a certain fixed focus pyrometer that the reading at 100 cm from a large source was but 70 per cent of that obtained when the instrument was mounted close to the source. For a certain focusable pyrometer the reading at 250 cm distance was 50° C different from the reading at 100 cm distance. These differences do not arise from the use of a source of insufficient size. In all cases the size of source was greater than that demanded by the geometry of the instrument, as described in the section on radiation pyrometry. In later instruments the construction has been modified to reduce these errors as much as possible. It is, however, advisable to employ approximately the same size of source and sighting distance in standardization and use of radiation pyrometers, or, if this can not be done, the same ratio of diameter of source to sighting distance.

(*a*) PRIMARY STANDARDIZATION.—In a primary standardization the radiation pyrometer is sighted into a black body, the temperature of which may be measured by rare-metal thermocouples. Since radiation pyrometers require a much larger source than optical pyrometers, the forms of black body previously described are not suitable. Fig. 160 illustrates one type of black body. The radiator *D* is a hollow graphite cylinder 16 by 8 cm, containing three diaphragms. This is mounted in an electrically-heated, platinum-wound, tube furnace 60 by 8 cm, with the back wall of the radiator 40 cm from the front of the furnace. The thermocouples *E* and *F* are protected by highly refractory porcelain tubes glazed on the outside. The furnace is inclined at an angle of about 30° to minimize air circulation. At *B* is located a water-cooled diaphragm of the proper opening. With the Fery pyrometer it is advisable to focus on the inmost opening *D* of the radiator. This requires special attention to insure that the front openings do not cut in on the cone of rays subtended by the pyrometer. The fixed-focus instruments

require so large a source that the front of the instrument must be within a few cm of the furnace. The diaphragms are accordingly cut with openings *increasing* in size toward the interior of the furnace, to fit the cone of rays illustrated in Figs. 66 and 68. Graphite oxidizes rapidly at high temperatures and also contaminates the furnace, so that it is sometimes desirable to have the black body made of refractory porcelain.

(*b*) SECONDARY STANDARDIZATION.—The use of an electric furnace and black body of the type described has certain disadvantages. Several hours are required to obtain satisfactory temperature equilibrium, and the source is too small for many instruments. Once having obtained a primary calibration with a standard radiation pyrometer, instruments of the same type

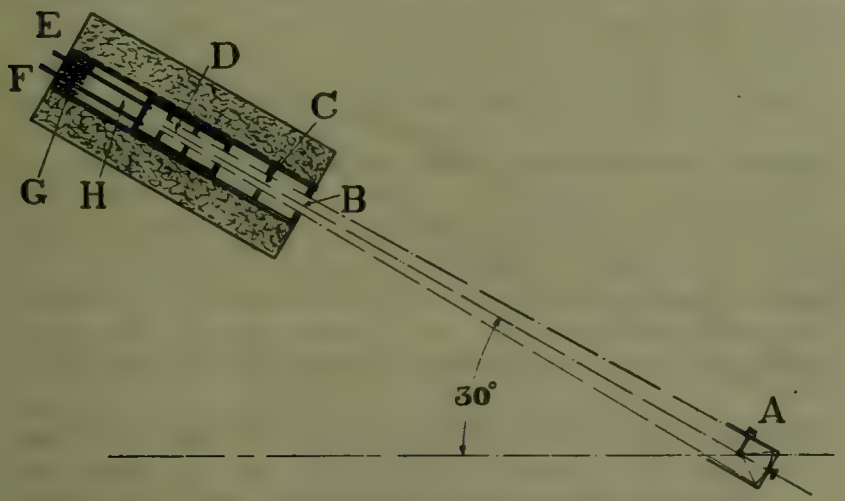


FIG. 160.—*Graphite black body and furnace for radiation pyrometers*

may be compared by sighting upon a uniformly heated source which is not emitting black-body radiation. A suitable source is obtained by electrically heating sheet nickel in air. A tough coat of nickel oxide forms which does not readily scale, provided the cooling from 500° C to room temperature is done slowly. A strip 17 cm long (exposed section), 13 cm wide, and 0.015 cm thick furnishes a circular area of 12 cm in diameter which is uniform in temperature to within 2° at 1200° C. A blackened water-cooled diaphragm having an opening of the required size is mounted directly in front of the glowing strip, and the pyrometer is sighted upon this opening. Readings are made alternately with the standard instrument and the pyrometer under test.

(1) *Computation of Standardization Data.*—The method of computation of observed data is the same with either the primary or secondary standardization. The relation between emf e and the absolute temperature ϑ is of the form $e = a\vartheta^b$, where b is approximately 4. This must be considered as a purely empirical equation. The fact that its form was suggested by the Stefan-Boltzman law is incidental, because there are many reasons why the latter law is not strictly applicable. The emf of the couple is not exactly proportional to the difference in temperature of the hot and cold junctions, and the temperature of the hot junction

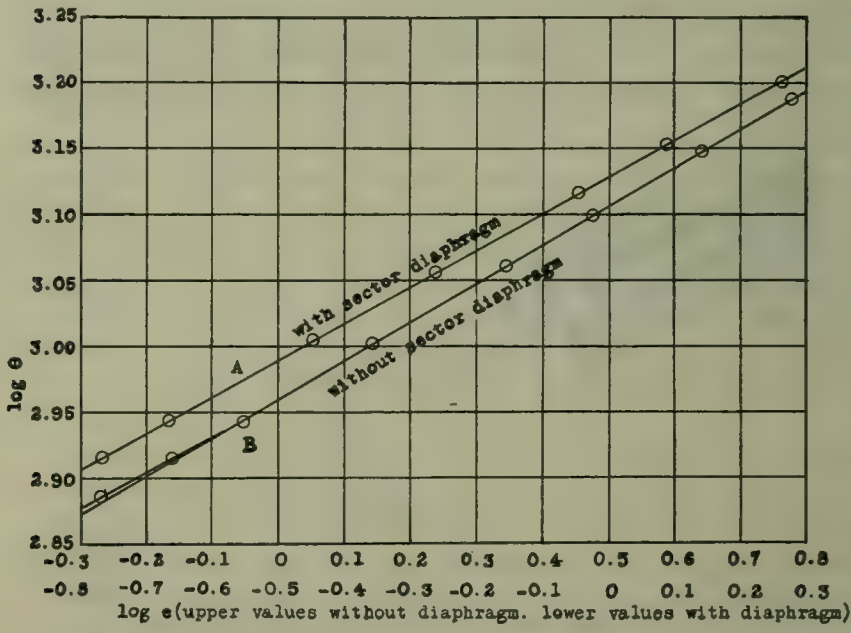


FIG. 161.—*Calibration plot for radiation pyrometer*

is not proportional to the rate at which radiation is absorbed. With increasing temperature of the source the temperature of the receiver increases considerably in some of the Fery pyrometers, rising to 100° C above that of the room, thus introducing a complicated variation in conduction and convection losses. Furthermore, the total reflection coefficient of the mirrors depends upon the temperature of the source. Hence, the justification of the above equation lies in the fact that it appears to satisfy the observations over a considerable temperature range. The empirical relation has been extended to apply to lower temperatures by the addition of a ϑ_o term, as follows: $e = a\,(\vartheta^b - \vartheta_o{}^b)$; but, in

general, the simpler form is sufficient for the temperature ranges ordinarily employed. Accordingly, expressed in the logarithmic form, the equation for the radiation pyrometer becomes:

$$\text{Log } e = \log a + b \log \vartheta.$$

The observed data are plotted log e versus log ϑ and the best straight line is drawn through the points, as shown by Fig. 161. At the lowest temperatures there may be a slight tendency for the observed points to lie above this line, in which case the data may be given weight by allowing the line to curve a little in this interval.

The sectored diaphragm used for extending the temperature range of the Fery pyrometer should reduce the amount of radiation falling upon the receiver by a constant factor, and hence it might be expected that if $e = a\,\vartheta^b$ represents the calibration without the diaphragm, $e = Ka\vartheta^b$, where K is a constant, would represent the calibration with the diaphragm. Thus, on the log plot the two straight lines would be parallel. Frequently, however, the lines best satisfying the data are not exactly parallel, as shown by Fig. 161. A table or curve of emf versus temperature is prepared from the values obtained in the above manner.

In work of high precision the emfs are measured with a potentiometer. If a galvanometer graduated in temperature is employed, the following method may be observed in determining the corrections to the scale at the higher range of temperature. Suppose the galvanometer is graduated from 600 to 1300° C, using the pyrometer without the sector and from 1300 to 2000° C with the sector. The highest calibration point is 1400° C, which is not sufficient to determine directly the scale error in the high range.

Accordingly, the instrument with the sectored diaphragm is calibrated from 600 to 1400° C, the emf being measured by a potentiometer. The data are plotted log e versus log ϑ and the curve linearly extrapolated to 2000° C. The galvanometer high-range scale versus emf calibration is made as discussed under Fig. 152, the resistance x being equal to that of the lead wires and couple of the radiation pyrometer. A table is made of true temperature versus emf of the instrument and of temperature scale of the galvanometer versus emf. These are compared at equal emfs, giving directly a table of temperature scale versus true temperature throughout the high range.

8. STANDARDIZATION OF RESISTANCE THERMOMETERS

If an accurately calibrated resistance-measuring instrument is employed, the most precise method of standardizing a pure platinum resistance thermometer is by observations of the resistance at the temperatures of melting ice, steam, and sulphur vapor.

(*a*) ICE POINT.—A jar or double-walled vacuum flask about 30 cm deep is filled with finely divided ice thoroughly wet with distilled water. The ice should reach to the bottom of the flask. The resistance R_0 of the thermometer immersed in this bath is determined.

(*b*) STEAM POINT.—The Regnault hypsometer affords a satisfactory steam bath. With slow boiling, except for work of the highest precision, the pressure of the steam may be assumed the same as that of the outside air. The temperature is obtained from the following formula where p denotes the barometric pressure in mm of Hg:

$$t°C = 100 + 0.03670\ (p - 760) - 0.00002046\ (p - 760)^2.$$

(*c*) SULPHUR BOILING POINT.—The following specifications have been prepared by Mueller and Burgess:[55]

All experimental work published has been drawn upon in preparation of these specifications. The specifications relate to apparatus and procedure suitable for the standardization primarily of resistance thermometers and thermocouples, and it was therefore considered permissible to limit the type of apparatus somewhat closely. The aim has been to impose conditions which are sufficient to insure that the thermometer shall assume a definite temperature, dependent only upon the pressure, when placed in the apparatus, and the question as to whether these conditions are really necessary has received only secondary consideration. Thus the question as to the temperature attained in tubes larger or smaller than those specified need not be considered. It may further be supposed that the thermometer or couple will be mounted in a protecting tube or sheath, and that the diameter of such a tube or width of the sheath will be less than 2 cm.

The specifications relate to the following matters: (1) Boiling apparatus, (2) purity of sulphur, (3) radiation shield, (4) procedure, and (5) computations.

(1) *Boiling Apparatus.*—The boiling tube is of glass, fused silica or similar material, and has an internal diameter of not less than 4 or more than 6 cm. The length must be such that the length of the vapor column measured from the surface of the liquid sulphur to the level of the top of the insulating material surrounding the tube shall exceed the length of the thermometer coil by at least 20 cm.[56]

Heating is by any suitable heater at the bottom of the tube, and the arrangement must be such that the heating element, and all conducting material in contact with it, terminate at least 4 cm below the level of the liquid sulphur. If a flame is allowed

[55] Mueller and H. A. Burgess, B. S. Sci. Papers No. 339.

[56] This length was arrived at as follows: The minimum distance from the liquid surface to the bottom of the shield was taken as 6 cm, excess of length of shield over length of thermometer coil 6 cm, distance available for displacing thermometer 6 cm, and minimum distance from top of shield to level of top of insulation 2 cm.

to impinge directly on the tube, the heat insulation must extend at least 4 cm below the level of the liquid sulphur. There should be a ring of insulating material above the heater, fitting the tube closely, to prevent superheating of the vapor by convection currents outside the tube. Above the heater the tube is surrounded with insulating material, not necessarily in contact with it, and of such character as to provide heat insulation equivalent to a thickness of not less than 1 cm of asbestos. The length of this insulation has already been specified. Any device used to close the top of the boiling tube must allow a free opening for equalization of pressure.

(2) *Purity of Sulphur.*—The sulphur should contain not over 0.02 per cent of impurities. It should be tested to determine whether selenium is present.

(3) *Radiation Shield.*—The radiation shield consists of a cylinder open at both ends and provided with a conical umbrella above. The cylindrical part is to be 1.5 to 2.5 cm larger in diameter than the protecting tube of the thermometer, and at least 1 cm smaller than the inside diameter of the boiling tube. The cylinder should extend 1.5 cm or more beyond the coil at each end. The umbrella should fit the thermometer tube closely, should overhang the cylinder, and be separated from the latter by a space 0.5 to 1 cm high. The inner surface of the cylinder must be a poor reflector, such as sheet iron, blackened aluminum, asbestos, or a deeply corrugated surface.

(4) *Procedure.*—The sulphur is brought to boiling [57] and the heating is so regulated that the condensation line is sharply defined and is 1 cm or more above the level of the top of the insulating material. The thermometer, inclosed in its shield, is inserted into the vapor, taking care to have the thermometer coil properly located with respect to the shield, and the thermometer and shield centered in the boiling tube. After putting the thermometer into the vapor time must be allowed for the line of condensation again to reach its proper level. Simultaneous readings of the temperature and barometric pressure are then made. In all cases care should be taken to prove that the temperature is not affected by displacing the thermometer 2 or 3 cm either up or down from its usual position.

(5) *Computations.*—Temperatures are calculated from the pressure by use of the formula,

$$t°\text{C}=444.°60+0.0910\,(p-760)-0.000049\,(p-760)^2.$$

If necessary, account should be taken of any difference in pressure between the levels at which the thermometer bulb and the open end of the barometer, respectively, are located. Pressures are to be expressed in the equivalent millimeters of mercury at 0° and under standard gravity (g=980.665).

Fig. 162 shows a gas-heated sulphur boiling-point apparatus and radiation *shield*. Electrically heated baths, properly constructed, are more satisfactory.[58]

[57] If the sulphur has been allowed to solidify in the bottom of the tube, it must be melted from the top downward to avoid breaking the tube. A better procedure is that recommended by Rothe (Z. S. für Instrk., 23, p. 366; 1903), viz, on completing work with the apparatus it is turned so that the tube makes an angle of 30° or less with the horizontal, so that the sulphur on solidifying extends along the sides of the tube, in which position it may be melted down with less danger of breaking the tube. Even when the procedure recommended is followed, breakage of tubes may be reduced by carefully melting the sulphur from the top downward over a bunsen burner before applying heat to it in the apparatus.

[58] Mueller and H. A. Burgess, B. S. Sci. Papers No. 339.

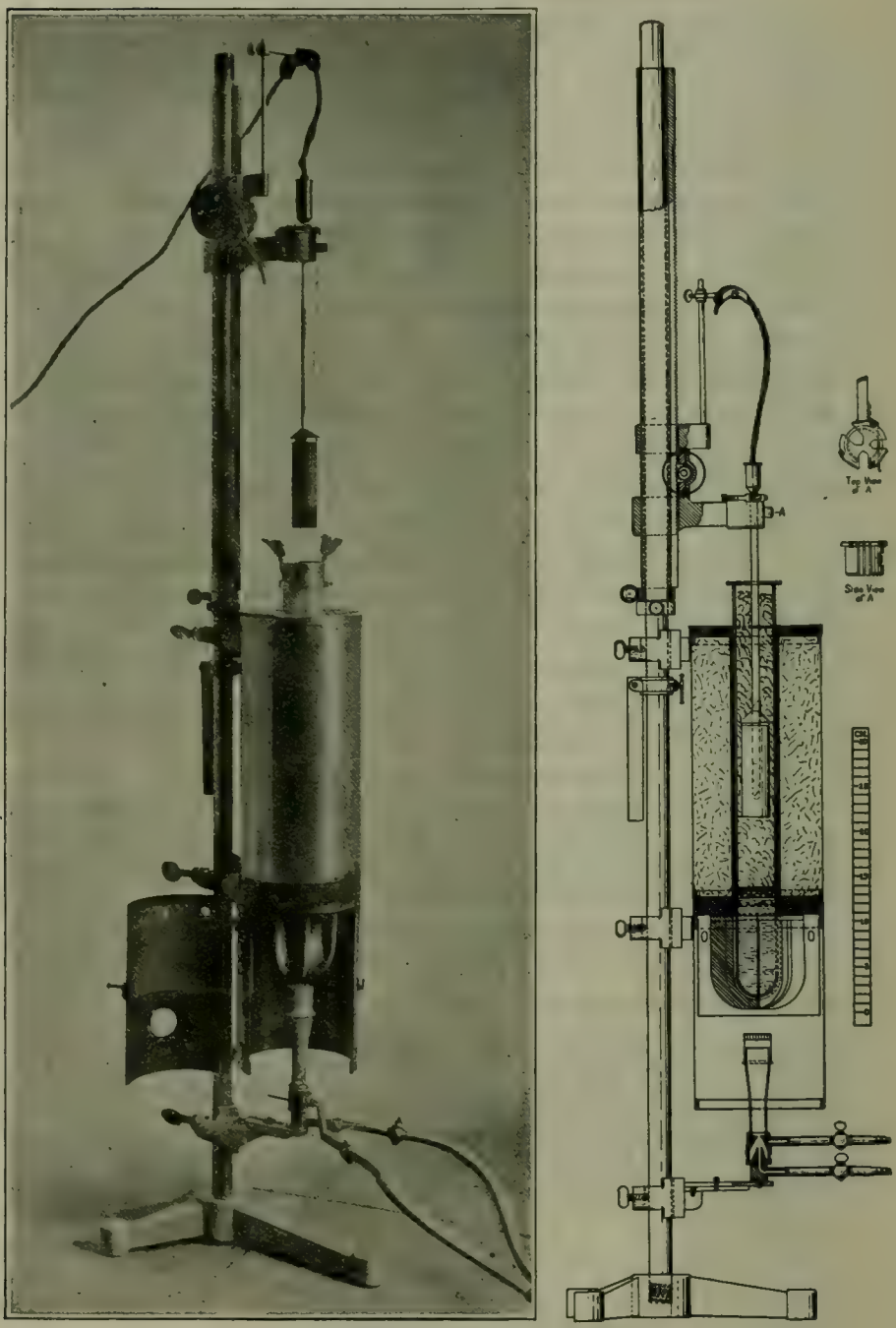


FIG. 162.—*Sulphur boiling-point apparatus*

(*d*) COMPUTATION OF DATA.—The relation between temperature t and resistance R is expressed by the following equations where pt is known as the "platinum temperature" and δ is a constant of value 1.48 to 1.52.

$$t - pt = \delta\left[\left(\frac{t}{100}\right)^2 - \frac{t}{100}\right]$$

$$pt = \frac{R - R_0}{R_{100} - R_0}\,100$$

From the resistance R_0 at 0° C and the observed resistance at the steam point, R_{100} is calculated as follows: From the accompanying table of t versus pt, the value of pt, corresponding to the actual steam temperature, is obtained. This and the observed resistance R is substituted in the second equation above, which is solved for R_{100}.

Values of t and pt

t° C	pt
99.000	99.015
99.400	99.409
99.800	99.803
100.000	100.000
100.200	100.197
100.600	100.591
101.000	100.985

The quantity δ is determined by substituting in the first formula for t and R their respective values at the sulphur-boiling point. The equations may be then computed and a table of R as a function of t prepared. This represents the standard temperature scale by definition, from −40 to 450° C, and is in agreement with the standard temperature scale up to 1100° C as closely as can be determined at the present time.

(*e*) NICKEL COILS.—Nickel is occasionally employed for use below 300° C. The equation $\log R = a + mt$ holds approximately from 0 to 300° C. Two standardization points, not too close together, are required for the determination of the constants a and m.

(*f*) THERMOMETERS WITH INDICATING INSTRUMENTS.—It is frequently desirable, especially for technical work in the industries, to standardize a thermometer and indicator as a unit. The precision of the ordinary indicator is usually not sufficient to require

primary standardization of the form described. A secondary method of standardization by comparing with other instruments, thermocouples, etc., in a uniformly heated electric furnace may be employed, the general procedure being that described in the section on standardization of couples. If, however, the laboratory is equipped for primary determinations, the following method is easier and more satisfactory. The relation $R=f(t)$ for the thermometer alone is determined from the ice, steam, and sulphur-point measurements. The indicator alone is calibrated at several points by connecting, in the place of the thermometer, known resistances and making the usual adjustments as for temperature measurements. These measurements may be accordingly so computed that a table of corrections to the reading of the indicator, when used with the thermometer, is obtained.

Instruments of the type shown in Fig. 77 should be standardized with leads of the same resistance as that employed in the use of the thermometer. Indicators of the type shown in Fig. 74 are calibrated by connecting the resistance standards between the points *d* and *e*.

Some of the strands of flexible leads may become broken, thus altering the resistance of the line and introducing an error of serious importance in precision measurements. Test for such a fault may be made by interchanging leads *CC'* and *TT'*, Fig. 74, or replacing the leads by another set in Fig. 77. Bad contacts and corroded lead wires and soldered junctions, etc., occasion considerable difficulty in industrial installations and must be always guarded against.

Further details on precision standardization of resistance thermometers are given in the papers referred to below.[59]

XII. INDUSTRIAL APPLICATIONS OF PYROMETRY

In the following paragraphs are considered several specific applications of pyrometry in the technical industries. This treatment is by no means intended to comprehensively review all compiled data in so extended a field, nor are the most important applications necessarily considered. The more or less obvious applications to temperature control of furnaces for annealing, ordinary heat treating, etc., are omitted entirely.

[59] Waidner and Burgess, B. S. Sci. Papers No. 124. Mueller and H. A. Burgess, B. S. Sci. Papers No. 339. Mueller, B. S. Sci. Papers No. 288.

1. GLASS MANUFACTURE

(*a*) TEMPERATURE MEASUREMENTS IN GLASS MELTING.—In the manufacture of glasses of lower melting point, such as bottle glasses, it has been customary to estimate the proper working conditions and indirectly the temperature by observing the physical and chemical properties of the mix in different parts of the furnace. Since the complete removal of bubbles, striæ, and chemical impurities in ordinary glassware is not always essential, accurate temperature control has not been considered necessary. There is no question, however, but that the application of pyrometry to the melting of ordinary glass is desirable. For the melting of optical glasses accurate control is one of the most important

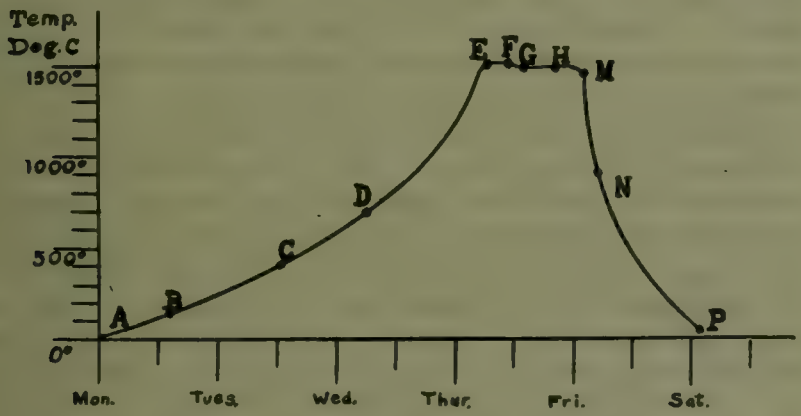


FIG. 163.—*Heating and cooling curve of a pot of optical glass*

factors affecting the quality of the glass. For example, there is a rather narrow range of temperature above which the increased solution of the pot material results in coloring of the glass and in leakage from the pot, and below which the glass does not fine successfully, but becomes milky and full of bubbles. If the temperatures are not reproduced from time to time the volatilization and solution are not constant, thus resulting in changes of composition and optical properties of the various batches. The proper heat treatment must be determined for each type of glass

Fig. 163 shows a complete heating and cooling of a particular pot of optical glass.[60] After the clay pot is molded and dried it is fired very slowly to prevent cracking and to a high temperature so that the sintering increases the strength. The firing takes place from *A* to *F*. From *G* to *H* the batch is introduced in several

[60] Keuffel, Chicago Pyrometry Symposium.

charges. From *H* to *M* the glass is thoroughly mixed by a clay stirring rod. It is then cooled and the stirring continued to *N*, during which time the bubbles and striæ are removed. At *N* the glass is so viscous that it can no longer be stirred. From *N* to *P* the glass is cooled sufficiently rapidly to break into chunks of the proper size. During the part of the curve represented by *G H M N* accurate control of the temperature is most essential.

On account of the flame in a tank furnace, a thermocouple placed at any point above the glass indicates the temperature of the hot gases surrounding it and not that of the glass. In such positions, pyrometers may often serve for general furnace control. The large differences in temperature in the molten glass and the crude process of tank-melting make it questionable whether a measure of the temperature of a particular portion of the glass affords any serviceable indication of temperature conditions. A thermocouple placed at the working end, especially in one of the arrangements used for removing glass at a definite rate, is valuable in regulating the temperature, and hence the viscosity of this portion of the glass. But the outstanding difficulty at present is the corrosion and short life of the thermocouple-protecting tubes when placed in contact with glass or even the furnace atmosphere. The destruction of the pyrometer may be avoided by sighting an optical pyrometer into a closed-end tube inserted into the glass through a diagonal hole cut in the wall just above the glass surface. The life of a tube used with an optical pyrometer is much longer than when employed as a protection to a couple.

The readings of an optical pyrometer sighted directly on the glass surface in a tank furnace are unreliable on account of the reflection of light from the flames. However, fairly satisfactory black-body conditions are obtained by sighting on the intersection of the glass surface and the opposite wall. Another method is to float a refractory block on the glass close to the wall and sight into the angle formed by the wall and the block. These methods may be checked by sighting into a closed tube.

The temperatures in pot furnaces are more uniform, and reliable measurements may be made by sighting the optical pyrometer into the pot or in some cases on the back or side walls of the furnace. Covered or hooded pots afford almost ideal black-body conditions. In general, in order to obtain the best temperature reproducibility the optical pyrometer should be used in exactly the same manner from time to time; that is, it should be sighted on the same portion of the furnace or pot.

In some glass pot and tank furnaces rare-metal couples inserted in the wall, and often not projecting entirely through into the interior, have been employed. The advantage of such an installation lies in the fact that recorders may be used and a record of the entire temperature cycle is obtained. Even though the temperature indicated is not that of the glass, sufficient control is possible, and, if necessary, the departure of the thermocouple temperature from that of the glass may be determined by the optical pyrometer. However, the continued use of rare-metal couples in a glass furnace is very costly on account of deterioration.

(*b*) General Principles of Annealing.—The object of annealing is to remove the strain in glass, thus minimizing the danger of breakage, and in addition, for optical glass, to make the material optically homogeneous. Annealing is accomplished by heating the glass to a uniform temperature at which it is sufficiently soft to allow the strains to relax in a short time, and then cooling uniformly to room temperature and slowly enough so that no appreciable strains are introduced. The factors which should be known are the maximum allowable rate of heating, the annealing temperature, the time the glass is to be maintained at this temperature, and the temperature-time curve of cooling.

For each type of glass there exists a short temperature range, at the lower limit of which a considerable time is required for relieving stresses and at the upper limit the stresses vanish very rapidly. The upper limit is characterized by several phenomena, such as the rapid deformation of strips or rods of the glass *under load*, the rapid disappearance of the strain figures and colors revealed by polarized light, a thermal transformation marked on heating by an absorption of heat, and by a sudden increase in thermal expansion. While the deformation of the glass at the upper limit is rapid under load, the glass is by no means soft enough to produce a rounding of corners or a deformation of shape under its own weight in the time required for annealing.

Table 29 shows the temperatures of several optical glasses, made by the Bureau of Standards, at which the heat absorption and rapid change in thermal expansion take place on heating. For rapid removal of stresses temperatures 20 or 30° higher may be used, but in general it is preferable to anneal at a temperature about 20° lower, so that the cooling may be more rapid without introducing new strains.

TABLE 29.—Annealing Temperature of Optical Glasses Made by the Bureau of Standards

Glass	Temperature	Glass	Temperature
	°C		°C
Dense flint	460	Light crown	495
Medium flint	455	Borosilicate crown	515
Light flint	485	Light barium crown	575
Barium flint	520		

The glass is held at some definite temperature within the annealing range until the strain has been sufficiently reduced and is then cooled at a rate depending upon the kind of glass, nature of the ware, dimensions, etc. Although the lower the annealing temperature the shorter the time required to cool, this gain is offset by the longer time necessary to reduce the strain at the annealing temperature. The optimum annealing temperature and rate of cooling may be determined empirically by examining for strain samples of glass cooled according to definite procedures. Without seriously increasing the strain in the finished glass the rate of cooling may be doubled for every 10° C decrease in temperature until it reaches a value consistent with a factor of safety from breakage, when it should be constant. The rate should be approximately inversely proportional to the square of the thickness of the glass. During annealing the surface of the glass must have a uniform temperature; otherwise new strains are introduced.[61]

(*c*) Temperature Measurements in Annealing Ovens.—The measurement of temperature in annealing ovens should serve the purpose of determining the exact temperature of the glass and the uniformity of heating. A decrease of about 10° C in the annealing temperature requires about twice the time for the same degree of annealing. Hence comparatively greater precision and sensitiveness of temperature measurement, especially with optical glass, is required in glass annealing than in ordinary annealing processes. The temperature should be known to better than 5° C, and the sensitivity should be 1 or 2° C. A portable potentiometer or a potentiometer recorder and a sensitive thermocouple afford the best equipment. As the temperature usually remains below 650° C, a base-metal couple such as iron-constantan or preferably chromel-alumel in a calorized iron protecting tube is satisfactory.

The couples should be so located that the true temperature of the glass is being measured. Flames or cold blasts of air should

[61] For a detailed account of methods of annealing see Tool and Valasek, B. S. Sci. Papers No. 358.

not come in contact with the couple or the glass. In order to ascertain the uniformity of heating a number of couples, depending on the size of the oven or kiln, should be located at various positions. In a lehr a minimum of two couples should be installed, one near the hot end and the other near the cold end, mounted down through the arch. A recorder is especially desirable in glass annealing, since improper temperature regulation at any time during the cooling may introduce serious strains, and the record chart shows whether or not the entire process has been carried out according to specification.

2. ROTARY PORTLAND-CEMENT KILNS

The high heat loss in cement kilns may be considerably reduced by close adherence to temperature specifications, experimentally determined under best working conditions, and the uniformity of the product may be improved by a suitable pyrometric equipment.

For controlling kiln operation a pyrometer installed in the rear end is most useful. It will indicate (or record) heat losses and kiln conditions such as draft, feed, fuel supply, rings, shutdowns, and under normal conditions the slow rise or fall of average temperature in the kiln.

The pyrometer best suited for this installation consists of a chromel-alumel thermocouple protected in a chromel or nichrome tube and connected to the ordinary indicator or recorder, which must be carefully protected from dust. The couple is inserted through the rear housing with its hot junction near the axis of the kiln and 1 or 2 feet within the kiln. A 6 or 7 foot couple will usually suffice. It should be kept away from the feed pipe in order to avoid the cooling action and the bridge of caked dust which forms. This cake should be removed at frequent intervals and at a regular hour, for its removal will change the indicated temperature.

In dry-process plants temperatures of 600° to occasionally as high as 1100° C are obtained. In wet-process plants any thermocouple has a long life and requires little attention because of the low temperature existing in the rear end.

A thermocouple installed in the kiln stack will serve, but with somewhat less satisfaction, particularly when the leakage of cold air into the housing is unsteady. However with the shorter kilns the increase in life of the couple obtained by removing it to the stack may be an economic advantage.

For obtaining data on the temperature of the clinkering zone, which are of greatest value during radical changes of operation such as a new fuel or raw mix, the optical pyrometer alone is suitable. A thermocouple inserted through the kiln wall with its hot junction flush with the inner face is a possibility, but the method is attended with many difficulties and uncertainties; for example, the connection to the recorder must be made through two collector rings.

Temperatures in the clinkering zone range from 1200 to 1500° C, depending upon the composition of the raw material, length of kiln, and length of hot zone. The clinkering zone is heated uniformly over a few feet only, and measurements should be taken at the hottest portion.

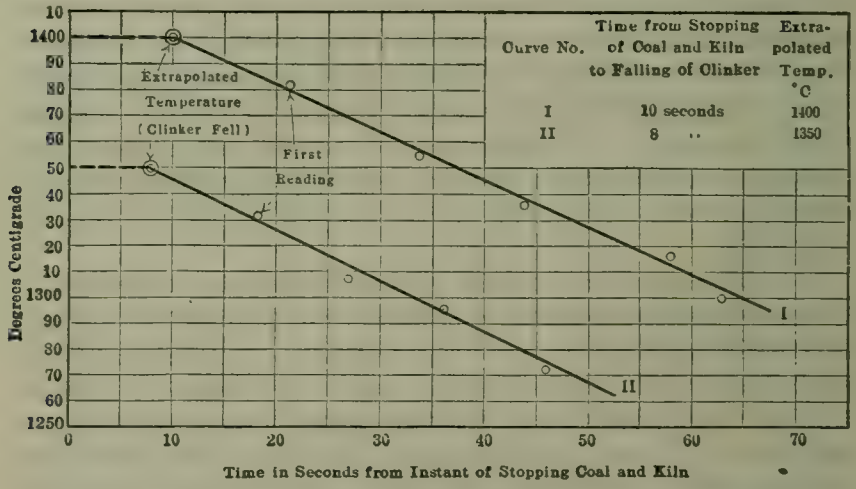

Curve No.
Time from Stopping of Coal and Kiln to Falling of Clinker
Extrapolated Temp. °C
I 10 seconds 1400
II 8 " 1350
Extrapolated Temperature (Clinker Fell)
First Reading
Degrees Centigrade
Time in Seconds from Instant of Stopping Coal and Kiln

FIG. 164.—*Typical extrapolation plot of temperature measurements on a rotary cement kiln*

These measurements are made with difficulty, and only a long series (taken, for example, every 30 minutes for 8 hours) is of value, as a single observation may be in gross error. The difficulties encountered are due to the presence of dust during operation and the high intrinsic brilliancy of the flame. Hence, in order to obtain useful measurements, it is necessary to stop the kiln and shut off the fuel and air. The optical pyrometer should be mounted in front of the kiln and sighted through an observation hole upon a spot on the inner wall which is uncovered as a clinker falls away. This spot is in the lower right-hand quadrant of the kiln, rotation being counterclockwise, facing the kiln. Upon stopping the kiln the clinker will soon fall, exposing the inner wall. It is not feasible

to sight on the mottled surface of the clinker, which cools with great rapidity as soon as the flame is cut off. The inner wall, however, does not cool materially until the clinker falls, and it presents a fairly uniform surface upon which to sight. On account of the great roughness of this surface and the surrounding hot walls, the apparent emissivity is high enough to make it unnecessary to apply a correction. A series of observations may be obtained within 1½ minutes, after waiting 5 to 15 seconds for the atmosphere to clear. Four or five readings should be made as rapidly as possible (every 10 seconds), the time of each reading being determined by a stop watch. These readings, plotted as in Fig. 164, show the temperature at the moment the clinker fell. A reading taken within 15 seconds from the time the clinker fell is not more than 30° lower than the temperature at zero time. The extrapolation, however, increases the accuracy if the observations are properly timed.[62]

3. CERAMIC INDUSTRIES

A brief historical review of the progress of pyrometry in the ceramic industries is of interest because at present there are in use both the most modern forms of temperature-measuring devices and the very old forms. Among the oldest of these is Wedgewood's contraction pyroscope. The principle of this method is still used in the present-day "Veritas firing system."[63] An improvement over Wedgewood's original pyroscope was announced in 1886 by Seger, who had constructed a series of fusible cones intended to indicate temperature intervals of 25° between 600 and 1800° C. Such cones are used in large quantities at present. They are known in this country as Seger cones, standard pyrometric cones, or Orton cones. They consist of small tetrahedra molded from various mixtures of kaolin, feldspar, lime, magnesia, quartz, iron oxide, lead oxide, and boric acid. Cones of higher number in the series have been developed to extend the range to 2000° (pure Al_2O_3 melts at 2050°). These are made by adding progressively greater amounts of alumina to kaolin. It is well known at present that the melting points of these cones do not correspond to definite temperatures.[64] However, the cone numbers have always been associated with temperature, the temper-

[62] Dana and Fairchild, Chicago Pyrometry Symposium.

[63] Cf. B. S. Tech. Papers No. 40: 1914.

[64] B. S. Tech. Papers Nos. 17 and 40. Sosman, Trans. Am. Ceram. Soc. 15, p. 482: 1913. Bureau of Mines Bull. No. 129; 1918.

atures given being in general higher than the final maturing temperature attained in a kiln heated to the corresponding cone number. The effect of time on the softening points of some cones is shown in Table 30, and the effects of oxidizing and reducing atmospheres is indicated in Table 31. These effects are greater and more irregular for cones melting in the lower ranges because of the admixtures of reducible and of volatile fluxes. The fact that the effects of time and of kiln gases on the softening of cones are somewhat similar to the effects of these variables on the progress of firing the ware is assigned as the reason for the successful and continued use of Seger cones. They do not give, however, a true measure of either of these effects, for they are never of the same material as the ware being fired. There is an advantage in favor of Veritas firing rings in this regard, since the latter may be made of the same mixture as that of the ware. Even in this case, however, there are differences readily apparent to the ceramist.

Neither Seger cones nor the firing rings are satisfactory gages of the efficiency obtained in firing, nor can they be used during drying, water smoking, or cooling. These faults have aided in the development and use of indicating and recording pyrometers. Nevertheless, the employment of cones will probably continue for many years, since they are inexpensive and can be placed at inaccessible points in a kiln as a check on the variation of heat conditions. Also their use requires a minimum of technical knowledge and training of the burner.

The application of pyrometers to processes in the ceramic industry has been retarded by the great complexity of the phenomena accompanying the vitrification of ceramic materials and the resultant difficulty in correlating temperature measurements with other variables, some of which are difficult to control or are unknown. It is just this condition which we believe will finally compel ceramists to depend entirely on pyrometers, especially recorders, to determine proper rates of firing and the best finishing temperatures. The pyrometer gives at least a true indication of temperature and temperature variation with time.

The types of pyrometers available for employment by the ceramic engineer are (1) thermocouple (preferably platinum, platinum-rhodium), (2) optical pyrometer, and (3) radiation pyrometer. There is, of course, a great variety of pyrometers and thermometers which can be used for the drying operation and

the low temperatures employed in enameling and decorating. The instruments are all described in detail in other sections.

(*a*) INSTALLATIONS.—In kilns fired between the temperatures 600 and about 1450° C the platinum, platinum-rhodium thermocouple is the most useful for recording the temperatures. This thermocouple protected with a porcelain tube and an extra fireclay or other outer tube is found to give satisfactory indications when the tip of the protection tube is extended into the kiln 3 to 4 inches beyond the inner face of the wall. Below 1300° C it is better to immerse the couple at least 6 inches, provided a sufficiently refractory tube is used. Above 1450° C adequate protection is very difficult and the general practice is to withdraw the couple and tubes until the tip is just flush with the inner face of the wall or even within the wall. In such a position obviously the lag is very great, and also the indicated temperature will be considerably below the correct value. Base-metal thermocouples, which can not be used above 1200° C, are more easily protected and should be inserted 6 inches or more beyond the inner wall. The best position to install the couple depends upon the type of kiln and its peculiar characteristics. Generally the couple is inserted through the roof, both in intermittent kilns and in continuous kilns of the tunnel and chamber types. In large kilns proportionately more couples are required. For example, in the tunnel kiln a couple installed every 20 feet of kiln length is advantageous. One of these should be in the hottest zone. Others should be opposite combustion chambers or grates, so that separate firing units may be controlled. In more complicated kilns, arranged to preheat air, to furnish heat for drying, etc., other couples are required. At present many manufacturers must do their own experimental work to determine the best points for installations. Portable outfits for this purpose are furnished by all pyrometer manufacturers.

The recording instrument is indispensable, and its great advantage over indicators is especially well recognized by ceramists. The most obvious advantage lies in the ability of the recorder to indicate plainly the occurrence of "setbacks" or otherwise the steady rise in temperature of intermittent kilns. This use alone is sufficient cause for the employment of pyrometers rather than cones. Multiple-point recorders are best for tunnel kilns and such intermittent kilns as require more than one couple. This recorder may confuse the records of a number of small kilns, making it desirable to use a simple recorder for each kiln.

Radiation pyrometers are of value when thermocouples can not be used for recording temperatures. Their use is discussed in detail in the section on radiation pyrometers.

The optical pyrometer, particularly the portable form, is useful in experimental work and may be employed to great advantage for routine adjustment of kilns to a uniform temperature. It logically replaces cones used for this purpose. "True" temperatures are easily obtained, and an exploration is made more quickly and accurately with this instrument than by observing the condition of cones. A fire-clay tube of 1 inch inside diameter inserted through the kiln wall and extending 6 to 12 inches within forms a very convenient means of measuring temperatures optically; otherwise readings may be obtained by sighting through a small opening onto the ware.

TABLE 30.—Softening Points of Cones, °C[a]

Cone	Rate of heating ° C/hour				Cone	Rate of heating ° C/hour			
	42.5	27.5	20.0	12.5		42.5	27.5	20.0	12.5
010	885	885	880	915	1	1100	1085	1090	1070
09	920	930	950	920	2	1110	1090	1100	1075
08	950	970	970	965	3	1120	1110	1110	1080
07	960	975	950	970	4	1130	1125	1115	1090
06	990	1000	990	995	5	1140	1135	1125	1110
05	1015	1035	1025	1030	6	1170	1140	1135	.....
04	1040	1055	1040	1045	7	1185	1155	1140	1125
03	1055	1065	1050	1050	8	1200	1170	1160	1150
02	1065	1070	1065	1060	9	1230	1190	1180	1190
01	1080	1080	1080	1070					

[a] Brown and Murray, B. S. Tech. Papers No. 17.

TABLE 31.—Softening Temperatures, °C, of Cones in Various Atmospheres[a]

Cone No.	Per cent of Fe_2O_3 in cone formula	Softening temperatures in—			Differences H_2-H_2O = $CO-CO_2$ atmospheres	H_2-H_2O and air
		Atmosphere of equal parts of H_2 and H_2O	Atmosphere of equal parts of CO and CO_2	Air		
06	8.52	1018	1026	1043	+ 8	+22
04	8.54	1089	1062	1085	−27	− 4
02	8.57	1119	1119	1178	0	+59
1	8.58	1146	1164	1182	−18	+36
2	4.38	1165	1170	1181	+ 5	+16
4	None	1221	1240	1250	+19	+29
6	None	1281	1292	1285	+11	+ 5
8	None	1332	1330	1365	− 2	+34
10	None	1358	1340	1334	−18	−24
12	None	1358	1370	1367	+12	+ 9

[a] Fieldner, Hall, and Field, Bur. Mines Bull. No. 129: 1918.

4. BY-PRODUCT COKE INDUSTRY

The measurement of the temperatures in and about a by-product coke oven is attended with special difficulties arising from inaccessibility of parts of the battery. Measurements may be taken at some points with comparative ease. If the construction engineer is informed regarding the desirability of making temperature measurements, he can readily provide the battery with openings for the insertion of thermocouples and for the use of the optical pyrometer:

(*a*) LOCATIONS FOR INSTALLING THERMOCOUPLES.—Measurements may be profitably made at the following points, which are usually accessible:

1. Waste-heat flue or tunnel at the end of the battery, and possibly on each side of the battery.

2. Heating walls (and recuperator walls) in the combustion flues or passages.

3. Regenerators, either individual or otherwise. The hot ends are usually inaccessible, but can very well be made accessible at some points.

4. Ascension or offtake pipes from the ovens.

5. In vapors and in coal in the ovens.

An inaccessible point, and one where probably more useful information can be obtained than at any other, is near the surface of the oven wall next to the coal. It is possible and wholly practicable to provide one or two ovens in a battery with holes in the walls about 3 cm in diameter and extending in about 2 meters from either the pusher side or coke side. Thermocouple protecting tubes are available which will not flux with silica brick below 1300° C, a temperature never intentionally reached at this point.

Thermocouples are commonly used only at point No. 1 above, but other installations have occasionally been made during experimental work. For properly controlling a battery the waste-heat thermoelectric pyrometer, and an optical pyrometer for measuring heating-wall temperatures are indispensable.

The thermocouples best suited for coke-oven work are the platinum, platinum-rhodium couples, and the chromel-alumel couples. For use in the vapors above the coal in the oven in the offtake pipe, and also in the waste-heat tunnel (under regenerative firing), any of the common types of base-metal couples excepting copper constantan are suitable. For permanent installation the

platinum couples should be of wire 0.5 or 0.6 mm in diameter, protected in glazed porcelain tubes of "Usalite" or "Impervite." The inner insulating tubes should be of the same material unglazed. This porcelain is easily cracked by sudden heating, and the successful insertion of the couple into a hot furnace is difficult to accomplish. There is no certainty of success without the use of a watch and a protection tube marked each centimeter of its length, so that the insertion may be made slowly and steadily. Ordinary tubes (walls 2 to 3 mm thick) may be thrust into a flue at 1200° C through a small opening without cracking, when they are inserted at a rate of about 5 cm per minute. Withdrawal should be made at a rate not greatly exceeding this, and a hot tube should not be allowed to touch cold objects other than poor conductors of heat, such as asbestos.

The waste-heat tunnel pyrometer should be installed as near the battery as possible and connected to a single-point recorder placed conveniently for the burner and foreman. Special attention should be given the cold junction of the couple. This should be buried underground or placed well above the tunnel roof where the temperature is not greatly influenced by the heat from the flue. Under regenerative firing waste gases are in the temperature range 200 to 350° C and vary 100° or less during reversal. The recorder should have a total scale range of 0 to 500° C. Records need not be made more frequently than once in two minutes; but, if it is desirable to connect this couple to a multiple-point recorder, more than one point on the recorder should be used, so that records are made at least every five minutes. A good thermocouple properly installed at this location will probably last for a number of years without serious deterioration.

In nonregenerative firing waste gases leave at about 1100° C, and a well-protected platinum couple must be used. It is best to employ the tubes mentioned above with an additional outer tube of fire clay, unglazed porcelain, etc., to protect the glazed porcelain from corrosion by the dust-laden gases. Fused quartz can not be used here.

(*b*) Measurements with the Optical Pyrometer.—An instrument with a scale range from 700 to 1400° C is required. A more extended range will seldom be used, since a temperature of 1400° C in the battery is dangerous. Heating wall temperatures can be easily measured in batteries having observation ports above each wall. The temperatures obtained by sighting on the nozzle

brick at the base of heating wall flues have a very remote connection with oven temperatures, but such measurements are of great utility for controlling the battery. Observations should be made in a flue in which there is no flame and when the nozzle opening is plainly visible. Attempts to obtain useful information by observations made on other points in the flue will generally be attended with failure because of the lack of black-body conditions, particularly in the upper part of the flue. However, temperature gradients *across* the battery may be adjusted by such observations.

Measurements with an optical pyrometer may be made at various other points in a battery where the temperature is over 700° C. It is difficult, but sometimes possible, to obtain a fairly good value for the temperature of the coke in the oven just before pushing the charge. This can be done only in ovens in which the top layer of coal is coked well enough to make it possible to clear the oven of smoke by removing a lid and opening the offtake pipe. Observations upon the coke during pushing as it emerges from the oven are practically impossible because of the very rapid cooling of the coke and the large clouds of smoke and dust which arise.

Observations made upon the oven walls after pushing are subject to very large errors on account of lack of black-body conditions, refraction of light by strong convection currents of air, and especially by the variation in emissivity of the oven face with changing layers of adhering coke.

(*c*) Experimental Measurements in Oven Proper.—There remains to be mentioned briefly the methods of employing couples within the coal charge during coking. One of the writers has successfully made a large number of such measurements, employing chromel-alumel thermocouples insulated with fire-clay nipples and protected in ordinary wrought-iron pipes. The latter were carefully inspected to see that there were no open seams, and before using they were thoroughly cleaned inside with dilute acid and dried. Such couples quickly deteriorate and the protection tubes must be removed after each charge. The maximum temperature attained is about 1100° C, and carbonization is rapid. The couples can be used for two charges. These thermocouples are inserted through holes in the oven lids, preferably in the center of the charge (midway between walls). Insertion can be made easily for a number of hours after charging, since the center of the charge remains at 100° C for half the coking period or longer. The heads of the couples must be very strong, as con-

siderable force is required in withdrawing the couples just before pushing. They can not be turned because the soft iron at 1100° will collapse.

Measurements in the coal are difficult, expensive, and have seldom been attempted. A discussion of the time-temperature relation obtained is beyond the scope of this paper. These measurements are more properly made in small laboratory furnaces and ovens, the opportunity afforded for research of practical utility being very promising.

5. STEEL MANUFACTURE

(*a*) BLAST FURNACE.—Temperature control of blast furnaces by pyrometric methods is in the experimental stage. Measurements with the optical pyrometer obtained by sighting through a glass window into a tuyère show temperatures from 1600° to above 1850° C. During tapping the temperature of the metal in the ordinary iron furnace varies from 1430 to 1475° C and that of the slag from 1430 to 1550° C. The method of observing these latter temperatures is described below.

(*b*) BESSEMER CONVERTER.—It is questionable whether the use of pyrometers will ever prove fruitful in the control of this process. Observations with the optical pyrometer on the apparent temperature of the flame are of little value. The temperature of the steel after blowing may be measured with the optical pyrometer by sighting on the liquid metal surface when the converter is tilted, and no corrections for emissivity are necessary, as approximate black-body conditions exist. The temperature during pouring may be obtained as described later.

(*c*) OPEN-HEARTH FURNACE—(1) *Dome.*—The temperature of the dome may be measured with the optical pyrometer by sighting on the inner surface of the brick, care being taken to avoid flames and fume. This is best done during reversals or at times when the flames are the least visible. The roof temperature varies from 1550 to 1750° C; but by means of pyrometric measurements the temperature may be maintained as close as possible to 1710° C, the melting point of the best silica brick.

(2) *Slag Pocket.*—Base-metal couples installed in the slag pocket assist in maintaining a proper supply of heat.

(3) *Slag Surface.*—The temperature of the slag surface in the furnace may be measured with the optical pyrometer, no correction for emissivity being necessary if the furnace temperature is fairly uniform. Care must be taken to avoid sighting through

brilliant flames. Slag temperatures are as much as 150° C above the temperature of the metal during heating. When the fire is shut off, the slag cools rapidly and may be 75° C below that of the metal just before tapping.

(4) *Metal Bath.*—Measurements of the temperature of the metal within the furnace have not been as yet reduced to routine practice, although much investigational work has been done.[65] One method is to immerse an Acheson graphite rod, block, or tube through the slag into the metal until it has attained temperature equilibrium and then raise the graphite above the slag surface and immediately observe its temperature with the optical pyrometer. The slag does not adhere, and no correction for emissivity is necessary. The method may be modified by removing the graphite quickly and observing the cooling curve in air and extrapolating the curve to the time at which the graphite was withdrawn from the bath. A similar method may be employed in spooning a sample of the metal, the optical pyrometer being sighted on the clear liquid surface. In this case the corrections for emissivity given in Table 16 should be made.

A few successful observations have been obtained by immersing a special refractory tube into the metal and sighting into the inside of the tube with the optical pyrometer. The tube was made of steel tipped with an Acheson graphite tube and protected by a fire-clay sleeve. Precautions must be taken to remove the smoke from the line of sight. All of these methods are inconvenient, but afford about the only means so far devised for obtaining the temperature of the metal before tapping.

(*d*) Tapping and Teeming.—The temperature of the clear metal in tapping and teeming may be easily and accurately measured by the optical pyrometer. The observer should stand from 10 to 20 feet from the stream and avoid sighting upon the slag, which appears as bright patches on the clear metal surface. Corrections for emissivity are applied as shown in Table 16. This table gives also the emissivity corrections for slag, temperature measurements of which are occasionally desirable.

(*e*) Rolling, etc.—The oxide formed on the surface of iron or steel heated in air readily scales and does not adhere well to the metal. This results in a film of air between the two surfaces and a considerable lowering of the outside temperature. It is accordingly necessary to remove the loosely adhering oxide before making a temperature measurement. In many processes, such as

[65] Cf. Chicago Pyrometry Symposium.

rolling, this is done automatically, and the optical pyrometer may be sighted directly upon the outside surface. This method has proved especially advantageous for steel rails while passing through the rolls.[66] Thus in four rail mills it was found that the ingot temperatures vary from 1075 to 1150° C, and the finishing temperatures of the rails from 880 to 1050° C. The measurements are capable of an accuracy of 10° C, so that close temperature specifications may be employed and greater uniformity of the product secured. The corrections for emissivity are usually negligible, but are given in Table 16.

The temperatures measured by the optical pyrometer are for the outside surface of the thin coat of oxide adhering after the flaky oxide is chipped off. There still exists a considerable temperature gradient through the remaining film. The relation between the temperatures of outside and inside surfaces of the oxide is apparently fairly well given by the following table.[67]

Temperatures, Degrees Centigrade

Outside of oxide layer	Inside of oxide layer
600	610
700	715
800	815
900	930
1000	1080

6. TEMPERATURES OF METAL SURFACES

The measurement of temperatures of metal surfaces is of importance in many investigational problems. If the surface is accessible and above a red heat, the optical pyrometer may be employed, corrections for emissivity being applied to the readings. At lower temperatures the radiation pyrometer might be employed, but, in general, it is unsatisfactory below 600° C. The most suitable method, especially for steam pipes, surfaces of stoves, etc., is by use of thermocouples. A special form of mounting must be made to reduce to a minimum the effect of heat conduction along the couple. Two small holes are drilled into the surface to a convenient depth and the bare end of each wire of the couple is peened into the separate holes. The ordinary welded junction is accordingly not employed, the circuit being closed through the metal surface. If the holes are drilled sufficiently close together that no serious temperature difference exists at the two points,

[66] Burgess, Rawdon, and Waltenberg, B. S. Tech. Papers, No. 38.
[67] Burgess and Foote, B. S. Sci. Papers No. 249

the introduction of a short length of iron, as a steam pipe, produces no effect. The temperature measured is that of the metal surface surrounding the point where the couple wires are peened, and if the wires are sufficiently small compared to the size of the surface the temperature of which is required, the conduction loss is negligible. This method has been employed in the measurement of heat transmission through boiler tubes,[68] and by this Bureau successfully for temperature measurements of steel girders embedded in concrete and subjected to fire. For problems of the latter type it affords about the only means for assuring that the temperature of the metal itself is being observed.

XIII. CONCLUSION

The rapid development in the application of pyrometry to the technical industries has been responsible in no small measure for the remarkable advancements characterizing all industrial processes requiring heat control.

The history of the application of pyrometric methods to technical processes dates from several centuries, but the major development has been effected in the past 20 years, and the rate of development is increasing.

A decade ago the word pyrometry required definition for the average being. Few, if any, technical schools considered the subject in their curricula. To-day pyrometric engineering is a profession, and one which promises unlimited opportunity.

With this recent growth of the industrial applications of pyrometry it is not surprising that the subject of pyrometry itself should have made rapid strides. New instruments are devised, new methods perfected, until at present a large pyrometric installation may be as extensive and intricate as a central telephone station.

This treatise has contained nothing of the historical, and the purely scientific aspects of pyrometry have been intentionally subordinated. It purports to describe practical methods in use in the industries for the year 1920. No doubt much will be antiquated in another half decade.

In conclusion, the writers desire to express their appreciation for the many valuable criticisms offered by their colleagues, especially Messrs. Waidner and Mueller, during the preparation of the manuscript.

WASHINGTON, February 11, 1920.

[68] Kreisinger and Barkley, Bur. Mines Tech. Paper No. 111

XIV. APPENDIX

CONTENTS

Page.

TABLE I.—Conversion table for degrees centigrade to degrees Fahrenheit (L. Waldo, Bull. A. I. M. E.; 1913) 301

TABLE II.—Conversion table for degrees Fahrenheit to degrees centigrade (L. Waldo, Bull. A. I. M. E.; 1913) 302

TABLE III.—Logarithms of tangents to tenth degree for use with Wanner pyrometers 304

TABLE IV.—Calibration data of representative couples 306

Fig. 165.—Chart showing various thermoelectric data 307

TABLE V.—Data of Geophysical Laboratory for Pt–90 Pt–10 Rh Thermocouple. 308

TABLE VI.—Data of Geophysical Laboratory for Copper—Constantan Thermocouple 309

Fig. 166 to Fig. 185.—Modern Pyrometric Installations 310

TABLE I.—Conversion Table Degrees Centigrade to Degrees Fahrenheit

C°	0	10	20	30	40	50	60	70	80	90
	F	F	F	F	F	F	F	F	F	F
−200	−328	−346	−364	−382	−400	−418	−436	−454	.....	.....
−100	−148	−166	−184	−202	−220	−238	−256	−274	−292	−310
−0	+32	+14	−4	−22	−40	−58	−76	−94	−112	−130
0	32	50	68	86	104	122	140	158	176	194
100	212	230	248	266	284	302	320	338	356	374
200	392	410	428	446	464	482	500	518	536	554
300	572	590	608	626	644	662	680	698	716	734
400	752	770	788	806	824	842	860	878	896	914
500	932	950	968	986	1004	1022	1040	1058	1076	1094
600	1112	1130	1148	1166	1184	1202	1220	1238	1256	1274
700	1292	1310	1328	1346	1364	1382	1400	1418	1436	1454
800	1472	1490	1508	1526	1544	1562	1580	1598	1616	1634
900	1652	1670	1688	1706	1724	1742	1760	1778	1796	1814
1000	1832	1850	1868	1886	1904	1922	1940	1958	1976	1994
1100	2012	2030	2048	2066	2084	2102	2120	2138	2156	2174
1200	2192	2210	2228	2246	2264	2282	2300	2318	2336	2354
1300	2372	2390	2408	2426	2444	2462	2480	2498	2516	2534
1400	2552	2570	2588	2606	2624	2642	2660	2678	2696	2714
1500	2732	2750	2768	2786	2804	2822	2840	2858	2876	2894
1600	2912	2930	2948	2966	2984	3002	3020	3038	3056	3074
1700	3092	3110	3128	3146	3164	3182	3200	3218	3236	3254
1800	3272	3290	3308	3326	3344	3362	3380	3398	3416	3434
1900	3452	3470	3488	3506	3524	3542	3560	3578	3596	3614
2000	3632	3650	3668	3686	3704	3722	3740	3758	3776	3794
2100	3812	3830	3848	3866	3884	3902	3920	3938	3956	3974
2200	3992	4010	4028	4046	4064	4082	4100	4118	4136	4154
2300	4172	4190	4208	4226	4244	4262	4280	4298	4316	4334
2400	4352	4370	4388	4406	4424	4442	4460	4478	4496	4514
2500	4532	4550	4568	4586	4604	4622	4640	4658	4676	4694
2600	4712	4730	4748	4766	4784	4802	4820	4838	4856	4874
2700	4892	4910	4928	4946	4964	4982	5000	5018	5036	5054
2800	5072	5090	5108	5126	5144	5162	5180	5198	5216	5234
2900	5252	5270	5288	5306	5324	5342	5360	5378	5396	5414
3000	5432	5450	5468	5486	5504	5522	5540	5558	5576	5594
3100	5612	5630	5648	5666	5684	5702	5720	5738	5756	5774
3200	5792	5810	5828	5846	5864	5882	5900	5918	5936	5954
3300	5972	5990	6008	6026	6044	6062	6080	6098	6116	6134
3400	6152	6170	6188	6206	6224	6242	6260	6278	6296	6314
3500	6332	6350	6368	6386	6404	6422	6440	6458	6476	6494
3600	6512	6530	6548	6566	6584	6602	6620	6638	6656	6674
3700	6692	6710	6728	6746	6764	6782	6800	6818	6836	6854
3800	6872	6890	6908	6926	6944	6962	6980	6998	7016	7034
3900	7052	7070	7088	7106	7124	7142	7160	7178	7196	7214
C°	0	10	20	30	40	50	60	70	80	90

C°	F°
1	1.8
2	3.6
3	5.4
4	7.2
5	9.0
6	10.8
7	12.6
8	14.4
9	16.2
10	18.0

F°	C°
1	.56
2	1.11
3	1.67
4	2.22
5	2.78
6	3.33
7	3.89
8	4.44
9	5.00
10	5.56
11	6.11
12	6.67
13	7.22
14	7.78
15	8.33
16	8.89
17	9.44
18	10.00

EXAMPLES: 1347° C. = 2444° F. + 12.6° F. = 2456.6° F. 3367° F. = 1850° C. + 2.78°.C. = 1852.78° C.

TABLE II.—Conversion Table Degrees Fahrenheit to Degrees Centigrade

(Dots denote recurring decimals)

F°	0	10	20	30	40	50	60	70	80	90
	C	C	C	C	C	C	C	C	C	C
—400	—240.0	—245.5	—251.1	—256.6	—262.2	—267.7	. . . .	. . . .	. . . .	. . . .
—300	—184.4	—190.0	—195.5	—201.1	—206.6	—212.2	—217.7	—223.3	—228.8	—234.4
—200	—128.8	—134.4	—140.0	—145.5	—151.1	—156.6	—162.2	—167.7	—173 3	—178.8
—100	—73.3	—78.8	—84.4	—90.0	—95.5	—101.1	—106.6	—112.2	—117 7	—123 3
—0	—17.7	—23.3	—28.8	—34.4	—40.0	—45.5	—51.1	—56.6	—62.2	—67.7
0	—17.7	—12.2	—6.6	—1.1	+4.4	+10.0	+15 5	+21.1	+26.6	+32.2
100	37.7	43.3	48.8	54.4	60.0	65.5	71.1	76.6	82.2	87 7
200	93.3	98.8	104.4	110.0	115.5	121.1	126.6	132.2	137.7	143.3
300	148.8	154.4	160.0	165.5	171.1	176.6	182.2	187.7	193.3	198.8
400	204.4	210.0	215 5	221.1	226.6	232.2	237.7	243.3	248.8	254.4
500	260.0	265.5	271.1	276.6	282.2	287.7	293.3	298.8	304.4	310.0
600	315.5	321.1	326.6	332.2	337.7	343.3	348.8	354.4	360.0	365.5
700	371.1	376.6	382.2	387.7	393.3	398.8	404.4	410.0	415.5	421.1
800	426.6	432.2	437.7	443.3	448.8	454.4	460.0	465.5	471.1	476.6
900	482.2	487.7	493.3	498.8	504.4	510.0	515.5	521.1	526.6	532.2
1000	537.7	543.3	548.8	554.4	560.0	565.5	571.1	576.6	582.2	587.7
1100	593.3	598.8	604.4	610.0	615.5	621.1	626.6	632.2	637.7	643 3
1200	648.8	654.4	660.0	665.5	67 .1	676.6	682.2	687.7	693.3	698.8
1300	704.4	710.0	715.5	721.1	726.6	732.2	737.7	743.3	748.8	754.4
1400	760 0	765.5	771.1	776.6	782.2	787.7	793.3	798.8	804.4	810.0
1500	815.5	821.1	826.6	832.2	837.7	843.3	848.8	854.4	860.0	865.5
1600	871.1	876.6	882.2	887.7	893.3	898.8	904.4	910.0	915.5	921.1
1700	926.6	932.2	937.7	943.3	948.8	954.4	960.0	965.5	971 1	976.6
1800	982.2	987.7	993.3	998.8	1004.4	1010.0	1015.5	1021.1	1026.6	1032.2
1900	1037.7	1043.3	1048.8	1054.4	1060.0	1065.5	1071.1	1076.6	1082.2	1087.7
2000	1093.3	1098.8	1104.4	1110.0	1115.5	1121.1	1126.6	1132.2	1137.7	1143.3
2100	1148.8	1154.4	1160.0	1165.5	1171.1	1176.6	1182.2	1187.7	1193.3	1198.8
2200	1204.4	1210.0	1215.5	1221.1	1226.6	1232.2	1237.7	1243.3	1248.8	1254.4
2300	1260.0	1265.5	1271.1	1276.6	1282.2	1287.7	1293.3	1298.8	1304.4	1310.0
2400	1315.5	1321.1	1326.6	1332.2	1337.7	1343.3	1348.8	1354.4	1360.0	1365.5
2500	1371.1	1376.6	1382.2	1387.7	1393.3	1398.8	1404.4	1410.0	1415.5	1421.1
2600	1426.6	1432.2	1437.7	1443.3	1448.8	1454.4	1460.0	1465.5	1471.1	1476.6
2700	1482.2	1487 7	1493.3	1498.8	1504.4	1510.0	1515.5	1521.1	1526.6	1532.2
2800	1537.7	1543.3	1548.8	1554.4	1560.0	1565.5	1571.1	1576.6	1582.2	1587.7
2900	1593.3	1598.8	1604.4	1610.0	1615.5	1621.1	1626.6	1632.2	1637.7	1643.3
3000	1648.8	1654.4	1660.0	1665.5	1671.1	1676.6	1682.2	1687.7	1693.3	1698.8
3100	1704.4	1710.0	1715.5	1721.1	1726.6	1732.2	1737.7	1743.3	1748.8	1754.4
3200	1760.0	1765.5	1771.1	1776.6	1782.2	1787.7	1793.3	1798.8	1804.4	1810.0
3300	1815.5	1821.1	1826.6	1832.2	1837.7	1843.3	1848.8	1854.4	1860.0	1865.5
3400	1871.1	1876.6	1882.2	1887.7	1893.3	1898.8	1904.4	1910.0	1915.5	1921.1
3500	1926.6	1932.2	1937.7	1943.3	1948.8	1954.4	1960.0	1965.5	1971.1	1976.6
3600	1982.2	1987.7	1993.3	1998.8	2004.4	2010.0	2015.5	2021.1	2026.6	2032.2
F°	0	10	20	30	40	50	60	70	80	90

F°	C°
1	0.5
2	1.1
3	1.6
4	2.2
5	2.7
6	3.3
7	3.8
8	4.4
9	5.0

EXAMPLES: —246.0° F. = —151.11° C. — 3.33° C. = —154.44° C.
3762° F. = 2071.11° C. + 1.11° C. = 2072.22° C.
2423.5° F. = 1326.666° C. + 1.666° C. + 0.277° C. = 1328.609° C.

TABLE II—Continued

°F to °C

F°	0	10	20	30	40	50	60	70	80	90
	C	C	C	C	C	C	C	C	C	C
3700	2037.7	2043.3	2048.8	2054.4	2060.0	2065.5	2071.1	2076.6	2082.2	2087.7
3800	2093.3	2098.8	2104.4	2110.0	2115.5	2121.1	2126.6	2132.2	2137.7	2143.3
3900	2148.8	2154.4	2160.0	2165.5	2171.1	2176.6	2182.2	2187.7	2193.3	2198.8
4000	2204.4	2210.0	2215.5	2221.1	2226.6	2232.2	2237.7	2243.3	2248.8	2254.4
4100	2260.0	2265.5	2271.1	2276.6	2282.2	2287.7	2293.3	2298.8	2304.4	2310.0
4200	2315.5	2321.1	2326.6	2332.2	2337.7	2343.3	2348.8	2354.4	2360.0	2365.5
4300	2371.1	2376.6	2382.2	2387.7	2393.3	2398.8	2404.4	2410.0	2415.5	2421.1
4400	2426.6	2432.2	2437.7	2443.3	2448.8	2454.4	2460.0	2465.5	2471.1	2476.6
4500	2482.2	2487.7	2493.3	2498.8	2504.4	2510.0	2515.5	2521.1	2526.6	2532.2
4600	2537.7	2543.3	2548.8	2554.4	2560.0	2565.5	2571.1	2576.6	2582.2	2587.7
4700	2593.3	2598.8	2604.4	2610.0	2615.5	2621.1	2626.6	2632.2	2637.7	2643.3
4800	2648.8	2654.4	2660.0	2665.5	2671.1	2676.6	2682.2	2687.7	2693.3	2698.8
4900	2704.4	2710.0	2715.5	2721.1	2726.6	2732.2	2737.7	2743.3	2748.8	2754.4
5000	2760.0	2765.5	2771.1	2776.6	2782.2	2787.7	2793.3	2798.8	2804.4	2810.0
5100	2815.5	2821.1	2826.6	2832.2	2837.7	2843.3	2848.8	2854.4	2860.0	2865.5
5200	2871.1	2876.6	2882.2	2887.7	2893.3	2898.8	2904.4	2910.0	2915.5	2921.1
5300	2926.6	2932.2	2937.7	2943.3	2948.8	2954.4	2960.0	2965.5	2971.1	2976.6
5400	2982.2	2987.7	2993.3	2998.8	3004.4	3010.0	3015.5	3021.1	3026.6	3032.2
5500	3037.7	3043.3	3048.8	3054.4	3060.0	3065.5	3071.1	3076.6	3082.2	3087.7
5600	3093.3	3098.8	3104.4	3110.0	3115.5	3121.1	3126.6	3132.2	3137.7	3143.3
5700	3148.8	3154.4	3160.0	3165.5	3171.1	3176.6	3182.2	3187.7	3193.3	3198.8
5800	3204.4	3210.0	3215.5	3221.1	3226.6	3232.2	3237.7	3243.3	3248.8	3254.4
5900	3260.0	3265.5	3271.1	3276.6	3282.2	3287.7	3293.3	3298.8	3304.4	3310.0
6000	3315.5	3321.1	3326.6	3332.2	3337.7	3343.3	3348.8	3354.4	3360.0	3365.5
6100	3371.1	3376.6	3382.2	3387.7	3393.3	3398.8	3404.4	3410.0	3415.5	3421.1
6200	3426.6	3432.2	3437.7	3443.3	3448.8	3454.4	3460.0	3465.5	3471.1	3476.6
6300	3482.2	3487.7	3493.3	3498.8	3504.4	3510.0	3515.5	3521.1	3526.6	3532.2
6400	3537.7	3543.3	3548.8	3554.4	3560.0	3565.5	3571.1	3576.6	3582.2	3587.7
6500	3593.3	3598.8	3604.4	3610.0	3615.5	3621.1	3626.6	3632.2	3637.7	3643.3
6600	3648.8	3654.4	3660.0	3665.5	3671.1	3676.6	3682.2	3687.7	3693.3	3698.8
6700	3704.4	3710.0	3715.5	3721.1	3726.6	3732.2	3737.7	3743.3	3748.8	3754.4
6800	3760.0	3765.5	3771.1	3776.6	3782.2	3787.7	3793.3	3798.8	3804.4	3810.0
6900	3815.5	3821.1	3826.6	3832.2	3837.7	3843.3	3848.8	3854.4	3860.0	3865.5
7000	3871.1	3876.6	3882.2	3887.7	3893.3	3898.8	3904.4	3910.0	3915.5	3921.1
7100	3926.6	3932.2	3937.7	3943.3	3948.8	3954.0	3960.0	3965.5	3971.1	3976.6
7200	3982.2	3987.7	3993.3	3998.8	4004.4	4010.0	4015.5	4021.1	4026.6	4032.2
7300	4037.7	4043.3	4048.8	4054.4	4060.0	4065.5	4071.1	4076.6	4082.2	4037.7
7400	4093.3	4098.8	4104.4	4110.0	4115.5	4121.1	4126.6	4132.2	4137.7	4143.3
7500	4148.8	4154.4	4160.0	4165.5	4171.1	4176.6	4182.2	4187.7	4193.3	4198.8
7600	4204.4	4210.0	4215.5	4221.1	4226.6	4232.2	4237.7	4243.3	4248.8	4254.4
7700	4260.0	4265.5	4271.1	4276.6	4282.2	4287.7	4293.3	4298.8	4304.4	4310.0
7800	4315.5	4321.1	4326.6	4332.2	4337.7	4343.3	4348.8	4354.4	4360.0	4365.5
7900	4371.1	4376.6	4382.2	4387.7	4393.3	4398.8	4404.4	4410.0	4415.5	4421.1
F°	0	10	20	30	40	50	60	70	80	90

F°	C°
1	0 5
2	1.1
3	1 6
4	2.2
5	2 7
6	3 3
7	3 8
8	4.4
9	5 0

TABLE III.—Logarithms of Tangents

(For use with Wanner Pyrometers)

Deg.	°.0	°.1	°.2	°.3	°.4	°.5	°.6	°.7	°.8	°.9
0°	−∞	$\bar{3}$.2419	$\bar{3}$.5429	$\bar{3}$.7190	$\bar{3}$.8439	$\bar{3}$.9409	$\bar{2}$.0200	$\bar{2}$.0870	$\bar{2}$.1450	$\bar{2}$.1962
1	$\bar{2}$.2419	$\bar{2}$.2833	$\bar{2}$.3211	$\bar{2}$.3559	$\bar{2}$.3881	$\bar{2}$.4181	$\bar{2}$.4461	$\bar{2}$.4725	$\bar{2}$.4973	$\bar{2}$.5208
2	$\bar{2}$.5431	$\bar{2}$.5643	$\bar{2}$.5845	$\bar{2}$.6038	$\bar{2}$.6223	$\bar{2}$.6401	$\bar{2}$.6571	$\bar{2}$.6736	$\bar{2}$.6894	$\bar{2}$.7046
3	$\bar{2}$.7194	$\bar{2}$.7337	$\bar{2}$.7475	$\bar{2}$.7609	$\bar{2}$.7739	$\bar{2}$.7865	$\bar{2}$.7988	$\bar{2}$.8107	$\bar{2}$.8223	$\bar{2}$.8336
4	$\bar{2}$.8446	$\bar{2}$.8554	$\bar{2}$.8659	$\bar{2}$.8762	$\bar{2}$.8862	$\bar{2}$.8960	$\bar{2}$.9056	$\bar{2}$.9150	$\bar{2}$.9241	$\bar{2}$.9331
5	$\bar{2}$.9420	$\bar{2}$.9506	$\bar{2}$.9591	$\bar{2}$.9674	$\bar{2}$.9756	$\bar{2}$.9836	$\bar{2}$.9915	$\bar{2}$.9992	$\bar{1}$.0068	$\bar{1}$.0143
6	$\bar{1}$.0216	$\bar{1}$.0289	$\bar{1}$.0360	$\bar{1}$.0430	$\bar{1}$.0499	$\bar{1}$.0567	$\bar{1}$.0633	$\bar{1}$.0699	$\bar{1}$.0764	$\bar{1}$.0828
7	$\bar{1}$.0891	$\bar{1}$.0954	$\bar{1}$.1015	$\bar{1}$.1076	$\bar{1}$.1135	$\bar{1}$.1194	$\bar{1}$.1252	$\bar{1}$.1310	$\bar{1}$.1367	$\bar{1}$.1423
8	$\bar{1}$.1478	$\bar{1}$.1533	$\bar{1}$.1587	$\bar{1}$.1640	$\bar{1}$.1693	$\bar{1}$.1745	$\bar{1}$.1797	$\bar{1}$.1848	$\bar{1}$.1898	$\bar{1}$.1948
9	$\bar{1}$.1997	$\bar{1}$.2046	$\bar{1}$.2094	$\bar{1}$.2142	$\bar{1}$.2189	$\bar{1}$.2236	$\bar{1}$.2282	$\bar{1}$.2328	$\bar{1}$.2374	$\bar{1}$.2419
10°	$\bar{1}$.2463	$\bar{1}$.2507	$\bar{1}$.2551	$\bar{1}$.2594	$\bar{1}$.2637	$\bar{1}$.2680	$\bar{1}$.2722	$\bar{1}$.2764	$\bar{1}$.2805	$\bar{1}$.2846
11	2887	2927	2967	3006	3046	3085	3123	3162	3200	3237
12	3275	3312	3349	3385	3422	3458	3493	3529	3564	3599
13	3634	3668	3702	3736	3770	3804	3837	3870	3903	3935
14	3968	4000	4032	4064	4095	4127	4158	4189	4220	4250
15	$\bar{1}$.4281	$\bar{1}$.4311	$\bar{1}$.4341	$\bar{1}$.4371	$\bar{1}$.4400	$\bar{1}$.4430	$\bar{1}$.4459	$\bar{1}$.4488	$\bar{1}$.4517	$\bar{1}$.4546
16	4575	4603	4632	4660	4688	4716	4744	4771	4799	4826
17	4853	4880	4907	4934	4961	4987	5014	5040	5066	5092
18	5118	5143	5169	5195	5220	5245	5270	5295	5320	5345
19	5370	5394	5419	5443	5467	5491	5516	5539	5563	5587
20°	$\bar{1}$.5611	$\bar{1}$.5634	$\bar{1}$.5658	$\bar{1}$.5681	$\bar{1}$.5704	$\bar{1}$.5727	$\bar{1}$.5750	$\bar{1}$.5773	$\bar{1}$.5796	$\bar{1}$.5819
21	5842	5864	5887	5909	5932	5954	5976	5998	6020	6042
22	6064	6086	6108	6129	6151	6172	6194	6215	6236	6257
23	6279	6300	6321	6341	6362	6383	6404	6424	6445	6465
24	6486	6506	6527	6547	6567	6587	6607	6627	6647	6667
25	$\bar{1}$.6687	$\bar{1}$.6706	$\bar{1}$.6726	$\bar{1}$.6746	$\bar{1}$.6765	$\bar{1}$.6785	$\bar{1}$.6804	$\bar{1}$.6824	$\bar{1}$.6843	$\bar{1}$.6863
26	6882	6901	6920	6939	6958	6977	6996	7015	7034	7053
27	7072	7090	7109	7128	7146	7165	7183	7202	7220	7238
28	7257	7275	7293	7311	7330	7348	7366	7384	7402	7420
29	7438	7455	7473	7491	7509	7526	7544	7562	7579	7597
30°	$\bar{1}$.7614	$\bar{1}$.7632	$\bar{1}$.7649	$\bar{1}$.7667	$\bar{1}$.7684	$\bar{1}$.7701	$\bar{1}$.7719	$\bar{1}$.7736	$\bar{1}$.7753	$\bar{1}$.7771
31	7788	7805	7822	7839	7856	7873	7890	7907	7924	7941
32	7958	7975	7992	8008	8025	8042	8059	8075	8092	8109
33	8125	8142	8158	8175	8191	8208	8224	8241	8257	8274
34	8290	8306	8323	8339	8355	8371	8388	8404	8420	8436
35	$\bar{1}$.8452	$\bar{1}$.8468	$\bar{1}$.8484	$\bar{1}$.8501	$\bar{1}$.8517	$\bar{1}$.8533	$\bar{1}$.8549	$\bar{1}$.8565	$\bar{1}$.8581	$\bar{1}$.8597
36	8613	8629	8644	8660	8676	8692	8708	8724	8740	8755
37	8771	8787	8803	8818	8834	8850	8865	8881	8897	8912
38	8928	8944	8959	8975	8990	9006	9022	9037	9053	9068
39	9084	9099	9115	9130	9146	9161	9176	9192	9207	9223

TABLE III.—Continued

Logarithms of Tangents

Deg.	°.0	°.1	°.2	°.3	°.4	°.5	°.6	°.7	°.8	°.9
40°	$\bar{1}$.9238	$\bar{1}$.9254	$\bar{1}$.9269	$\bar{1}$.9284	$\bar{1}$.9300	$\bar{1}$.9315	$\bar{1}$.9330	$\bar{1}$.9346	$\bar{1}$.9361	$\bar{1}$.9376
41	$\bar{1}$.9392	$\bar{1}$.9407	$\bar{1}$.9422	$\bar{1}$.9438	$\bar{1}$.9453	$\bar{1}$.9468	$\bar{1}$.9483	$\bar{1}$.9499	$\bar{1}$.9514	$\bar{1}$.9529
42	$\bar{1}$.9544	$\bar{1}$.9560	$\bar{1}$.9575	$\bar{1}$.9590	$\bar{1}$.9605	$\bar{1}$.9621	$\bar{1}$.9636	$\bar{1}$.9651	$\bar{1}$.9666	$\bar{1}$.9681
43	$\bar{1}$.9697	$\bar{1}$.9712	$\bar{1}$.9727	$\bar{1}$.9742	$\bar{1}$.9757	$\bar{1}$.9772	$\bar{1}$.9788	$\bar{1}$.9803	$\bar{1}$.9818	$\bar{1}$.9833
44	$\bar{1}$.9848	$\bar{1}$.9864	$\bar{1}$.9879	$\bar{1}$.9894	$\bar{1}$.9909	$\bar{1}$.9924	$\bar{1}$.9939	$\bar{1}$.9955	$\bar{1}$.9970	$\bar{1}$.9985
45	0.0000	0.0015	0.0030	0.0045	0.0061	0.0076	0.0091	0.0106	0.0121	0.0136
46	0152	0167	0182	0197	0212	0228	0243	0258	0273	0288
47	0303	0319	0334	0349	0364	0379	0395	0410	0425	0440
48	0456	0471	0486	0501	0517	0532	0547	0562	0578	0593
49	0608	0624	0639	0654	0670	0685	0700	0716	0731	0746
50°	0.0762	0.0777	0.0793	0.0808	0.0824	0.0839	0.0854	0.0870	0.0885	0.0901
51	0916	0932	0947	0963	0978	0994	1010	1025	1041	1056
52	1072	1088	1103	1119	1135	1150	1166	1182	1197	1213
53	1229	1245	1260	1276	1292	1308	1324	1340	1356	1371
54	1387	1403	1419	1435	1451	1467	1483	1499	1516	1532
55	0.1548	0.1564	0.1580	0.1596	0.1612	0.1629	0.1645	0.1661	0.1677	0.1694
56	1710	1726	1743	1759	1776	1792	1809	1825	1842	1858
57	1875	1891	1908	1925	1941	1958	1975	1992	2008	2025
58	2042	2059	2076	2093	2110	2127	2144	2161	2178	2195
59	2212	2229	2247	2264	2281	2299	2316	2333	2351	2368
60°	0.2386	0.2403	0.2421	0.2438	0.2456	0.2474	0.2491	0.2509	0.2527	0.2545
61	2562	2580	2598	2616	2634	2652	2670	2689	2707	2725
62	2743	2762	2780	2798	2817	2835	2854	2872	2891	2910
63	2928	2947	2966	2985	3004	3023	3042	3061	3080	3099
64	3118	3137	3157	3176	3196	3215	3235	3254	3274	3294
65	0.3313	0.3333	0.3353	0.3373	0.3393	0.3413	0.3433	0.3453	0.3473	0.3494
66	3514	3535	3555	3576	3596	3617	3638	3659	3679	3700
67	3721	3743	3764	3785	3806	3828	3849	3871	3892	3914
68	3936	3958	3980	4002	4024	4046	4068	4091	4113	4136
69	4158	4181	4204	4227	4250	4273	4296	4319	4342	4366
70°	0.4389	0.4413	0.4437	0.4461	0.4484	0.4509	0.4533	0.4557	0.4581	0.4606
71	4630	4655	4680	4705	4730	4755	4780	4805	4831	4857
72	4882	4908	4934	4960	4986	5013	5039	5066	5093	5120
73	5147	5174	5201	5229	5256	5284	5312	5340	5368	5397
74	5425	5454	5483	5512	5541	5570	5600	5629	5659	5689
75	0.5719	0.5750	0.5780	0.5811	0.5842	0.5873	0.5905	0.5936	0.5968	0.6000
76	6032	6065	6097	6130	6163	6196	6230	6264	6298	6332
77	6366	6401	6436	6471	6507	6542	6578	6615	6651	6688
78	6725	6763	6800	6838	6877	6915	6954	6994	7033	7073
79	7113	7154	7195	7236	7278	7320	7363	7406	7449	7493
80°	0.7537	0.7581	0.7626	0.7672	0.7718	0.7764	0.7811	0.7858	0.7906	0.7954
81	0.8003	0.8052	0.8102	0.8152	0.8203	0.8255	0.8307	0.8360	0.8413	0.8467
82	0.8522	0.8577	0.8633	0.8690	0.8748	0.8806	0.8865	0.8924	0.8985	0.9046
83	0.9109	0.9172	0.9236	0.9301	0.9367	0.9433	0.9501	0.9570	0.9640	0.9711
84	0.9784	0.9857	0.9932	1.0008	1.0085	1.0164	1.0244	1.0326	1.0409	1.0494
85	1.0580	1.0669	1.0759	1.0850	1.0944	1.1040	1.1138	1.1238	1.1341	1.1446
86	1.1554	1.1664	1.1777	1.1893	1.2012	1.2135	1.2261	1.2391	1.2525	1.2663
87	1.2806	1.2954	1.3106	1.3264	1.3429	1.3599	1.3777	1.3962	1.4155	1.4357
88	1.4569	1.4792	1.5027	1.5275	1.5539	1.5819	1.6119	1.6441	1.6789	1.7167
89	1.7581	1.8038	1.8550	1.9130	1.9800	2.0591	2.1561	2.2810	2.4571	2.7581

TABLE IV.—Calibration Data of Representative Couples

Englehard "Le Chatelier"		Johnson-Matthey "Le Chatelier"		Copper-constantan		Iron-constantan			Chromel-alumel	
Emf	Temp., degrees C	Emf	Temp., degrees C	Emf	Temp., degrees C	Emf	[a] Temp., degrees C		Emf	Temp., degrees C
							B	*L*		
0	0	0	0	0	0	[illegible]	[illegible]	0	0	[illegible]
1	147	1	146	1	25	[illegible]	105	95	5	122
2	265	2	260	2	49	10	204	186	10	243
3	374	3	364	3	72	15	299	277	15	363
4	478	4	461	4	94	20	392	367	20	482
5	578	5	553	5	115	25	483	457	25	601
6	675	6	641	6	136	30	574	546	30	721
7	770	7	725	7	156	35	662	632	35	844
8	861	8	[illegible]	8	175	40	749	713	40	970
9	950	9	884	9	194	45	836	792	45	1100
10	1037	10	959	10	213	50	924	871	.....	.....
11	1122	11	1032	11	232	55	1011	950	.....	.....
12	1206	12	1103	12	250	60	.....	1030	.....	.....
13	1290	13	1173	13	268	.....	.....	.....	.....	.....
14	1373	14	1242	14	285	.....	.....	.....	.....	.....
15	1455	15	1311	15	302	.....	.....	.....	.....	.....
.....	.....	16	1379	16	319	.....	.....	.....	.....	.....
.....	.....	17	1447	17	336	.....	.....	.....	.....	.....
.....	.....	.....	.....	18	353	.....	.....	.....	.....	.....

[a] *B* represents mean calibration by U. S. Bureau of Standards of iron-constantan couples from all sources. *L* represents mean calibration of Leeds & Northrup's iron-constantan couple.

Explanatory note to Fig. 165.—

Emfs of various materials versus platinum, at different temperatures, the cold junctions being at zero °C. From this chart the emf of any combination of metals or alloys is found by measuring the vertical distance between the curves for the chosen materials, the upper curve indicating the positive element. Thus at 500° C the emf of a copel-platinum thermocouple would be 23.4 millivolts, copel being the negative element, while at the same temperature a platinum-chromel couple would develop 16 millivolts, chromel being positive to platinum and hence also positive to copel. The emf of a copel-chromel would be 16+23.4 millivolts at 500° C.

Where two curves are parallel in a given temperature range the emf of a couple made of the materials indicated would remain constant throughout that range. Where two curves cross, the emf of the couple indicated would decrease, become zero, and then increase in the reverse direction as the temperature is varied uniformly through this range, provided the cold junctions are at zero °C.

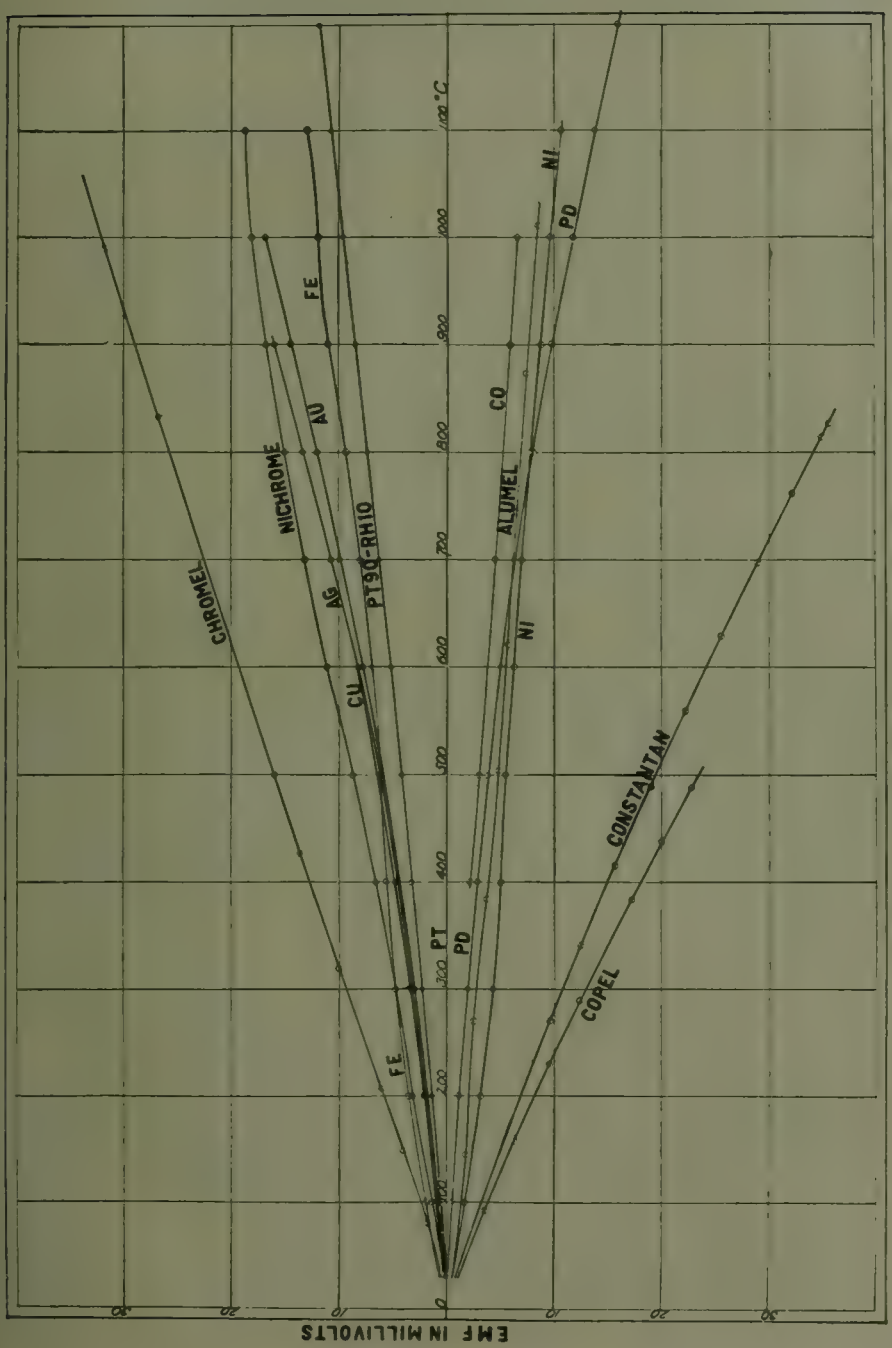


FIG. 105.—*Emfs of various materials versus platinum*

TABLE V.—Emf-temperature data for Pt-90 Pt 10 Rh Thermocouple as determined by the Geophysical Laboratory. (Adams, Bull. A. I. M. M. E., 159, p. 2111, 1919)

[Emfs are expressed in microvolts; temperatures in degrees centigrade; cold junction=0° C]

E μv	0	1000	2000	3000	4000	5000	6000
0	0 °C	147.1°C	265.4°C	374.3°C	478.1°C	578.3°C	675.3°C
	17.8	12.6	11.2	10.6	10.2	9.8	9.5
100	17.8	159.7	276.6	384.9	488.3	588.1	684.8
	16.7	12.4	11.1	10.5	10.1	9.8	9.5
200	34.5	172.1	287.7	395.4	498.4	597.9	694.3
	15.8	12.2	11.0	10.5	10.1	9.8	9.5
300	50.3	184.3	298.7	405.9	508.5	607.7	703.8
	15.1	12.0	11.0	10.4	10.1	9.7	9.5
400	65.4	196.3	309.7	416.3	518.6	617.4	713.3
	14.6	11.8	10.9	10.4	10.0	9.7	9.4
500	80.0	208.1	320.6	426.7	528.6	627.1	722.7
	14.1	11.6	10.9	10.4	10.0	9.7	9.4
600	94.1	219.7	331.5	437.1	538.6	636.8	732.1
	13.7	11.5	10.8	10.3	10.0	9.7	9.4
700	107.8	231.2	342.3	447.4	548.6	646.5	741.5
	13.4	11.5	10.7	10.3	9.9	9.6	9.4
800	121.2	242.7	353.0	457.7	558.5	656.1	750.9
	13.1	11.4	10.7	10.2	9.9	9.6	9.3
900	134.3	254.1	363.7	467.9	568.4	665.7	760.2
	12.8	11.3	10.6	10.2	9.9	9.6	9.3
1000	147.1	265.4	374.3	478.1	578.3	675.3	769.5

E μv	7000	8000	9000	10 000	11 000	12 000
0	769.5°C	861.1°C	950.4°C	1037.3°C	1122.2°C	1205.9°C
	9.3	9.0	8.8	8.6	8.4	8.3
100	778.8	870.1	959 2	1045.9	1130.6	1214.2
	9.2	9.0	8.8	8.5	8.4	8.4
200	788.0	879.1	968.0	1054.4	1139.0	1222.6
	9.2	9.0	8.7	8.5	8.4	8.3
300	797.2	888.1	976.7	1062.9	1147.4	1230.9
	9.2	9.0	8.7	8.5	8.4	8.4
400	806.4	897.1	985.4	1071.4	1155.8	1239.3
	9.2	9.0	8.7	8.5	8.4	8.3
500	815.6	906.1	994.1	1079.9	1164.2	1247.6
	9.1	8.9	8.7	8.5	8.3	8.3
600	824.7	915.0	1002.8	1088.4	1172.5	1255.9
	9.1	8.9	8.7	8.5	8.4	8.4
700	833.8	923.9	1011.5	1096.9	1180.9	1264.3
	9.1	8.9	8.6	8.5	8.3	8.3
800	842.9	932.8	1020.1	1105.4	1189.2	1272.6
	9.1	8.8	8.6	8.4	8.4	8.4
900	852.0	941.6	1028.7	1113.8	1197.6	1281.0
	9.1	8.8	8.6	8.4	8.3	8.3
1000	861.1	950.4	1037.3	1122.2	1205.9	1289.3

E μv	13 000	14 000	15 000	16 000	17 000	18 000
0	1289.3°C	1372.4°C	1454.8°C	1537.5°C	1620.9°C	1704.3°C
	8.4	8.3	8.2	8.3	8.3	8.3
100	1297.7	1380.7	1463.0	1545.8	1629.2	1712.6
	8.3	8.3	8.2	8.3	8.4	8.4
200	1306.0	1389.0	1471.2	1554.1	1637.6	1721.0
	8.3	8.3	8.2	8.3	8.3	8.3
300	1314.3	1397.3	1479.4	1562.4	1645.9	1729.3
	8.3	8.3	8.3	8.4	8.4	8.4
400	1322.6	1405.6	1487.7	1570.8	1654.3	1737.7
	8.3	8.2	8.3	8.3	8.3	8.3
500	1330.9	1413.8	1496.0	1579.1	1662.6	1746.0
	8.3	8.2	8.3	8.4	8.3	8.3
600	1339.2	1422.0	1504.3	1587.5	1670.9	1754.3
	8.3	8.2	8.3	8.3	8.4	
700	1347.5	1430.2	1512.6	1595.8	1679.3	
	8.3	8.2	8.3	8.4	8.3	
800	1355.8	1438.4	1520.9	1604.2	1687.6	
	8.3	8.2	8.3	8.3	8.4	
900	1364.1	1446.6	1529.2	1612.5	1696.0	
	8.3	8.2	8.3	8.4	8.3	
1000	1372.4	1454.8	1537.5	1620.9	1704.3	

TABLE VI.—Emf-temperature data for Copper-Constantan Thermocouple as determined by the Geophysical Laboratory. (Adams, Bull. A. I. M. M. E., 159, p. 2111, 1919)

[Emfs are expressed in microvolts; temperature in degrees centigrade; cold junction=0° C]

E μv	0	1000	2000	3000	4000	5000	6000
0	0	25. 27	49. 20	72. 08	94. 07	115. 31	135. 91
	2.59	2.45	2.33	2.23	2.16	2.09	2.03
100	2. 59	27. 72	51. 53	74. 31	96. 23	117. 40	137. 94
	2.57	2.43	2.32	2.23	2.15	2.08	2.02
200	5. 16	30. 15	53 85	76. 54	98. 38	119. 48	139. 96
	2.56	2.42	2.31	2.22	2.14	2.08	2.02
300	7. 72	32. 57	56. 16	78. 76	100. 52	121. 56	141. 98
	2.55	2.41	2.30	2.21	2.14	2.07	2.01
400	10. 27	34. 98	58. 46	80. 97	102. 66	123. 63	143. 99
	2.53	2.40	2.30	2.20	2.13	2.06	2.01
500	12. 80	37. 38	60. 76	83. 17	104. 79	125. 69	146. 00
	2.52	2.39	2.28	2.20	2.12	2.06	2.00
600	15. 32	39. 77	63. 04	85. 37	106. 91	127. 75	148. 00
	2.51	2.38	2.27	2.19	2.11	2.05	2.00
700	17. 83	42. 15	65. 31	87. 56	109. 02	129 80	150. 00
	2.49	2.36	2.27	2.18	2.10	2.04	1.99
800	20. 32	44. 51	67 58	89. 74	111 12	131. 84	151. 99
	2.48	2.35	2.25	2.17	2.10	2.04	1.99
900	22. 80	46. 86	69. 83	91. 91	113. 22	133. 88	153. 97
	2.47	2.34	2.25	2.16	2.09	2.03	1.98
1000	25. 27	49. 20	72. 08	94. 07	115. 31	135. 91	155. 95

E μv	7000	8000	9000	10 000	11 000	12 000	13 000
0	155. 95	175. 50	194. 62	213. 36	231. 74	249. 82	267. 60
	1.97	1.93	1.89	1.85	1.82	1.79	1.76
100	157. 92	177. 43	196. 51	215. 21	233. 56	251. 61	269. 36
	1.97	1.93	1.89	1.85	1.82	1.79	1.76
200	159. 89	179. 36	198. 40	217. 06	235. 38	253. 40	271. 12
	1.97	1.92	1.88	1.85	1.82	1.78	1.76
300	161. 86	181. 28	200. 28	218. 91	237. 20	255. 18	272. 88
	1.96	1.92	1.88	1.84	1.81	1.78	1.76
400	163. 82	183. 20	202. 16	220. 75	239. 01	256. 96	274. 64
	1.96	1.91	1.88	1.84	1.81	1.78	1.76
500	165. 78	185. 11	204. 04	222. 59	240. 82	258. 74	276. 40
	1.95	1.91	1.87	1.84	1.81	1.78	1.75
600	167. 73	187. 02	205. 91	224. 43	242. 63	260. 52	278. 15
	1.95	1.91	1.87	1.83	1.80	1.77	1.75
700	169. 68	188. 93	207. 78	226. 26	244. 43	262. 29	279. 90
	1.94	1.90	1.86	1.83	1.80	1.77	1.75
800	171. 62	190. 83	209. 64	228. 09	246. 23	264. 06	281. 65
	1.94	1.90	1.86	1.83	1.80	1.77	1.74
900	173. 56	192. 73	211. 50	229. 92	248. 03	265. 83	283. 39
	1.94	1.89	1.86	1.82	1.79	1.77	1.74
1000	175. 50	194. 62	213. 36	231. 74	249. 82	267. 60	285. 13

E μv	14 000	15 000	16 000	17 000	18 000	19 000
0	285. 13	302. 42	319. 49	336. 36	353. 08	369. 61
	1.74	1.72	1.70	1.68	1.66	1.64
100	286. 87	304. 14	321. 19	338. 04	354. 74	371. 25
	1.74	1.71	1.69	1.68	1.66	1.64
200	288. 61	305. 85	322. 88	339. 72	356. 40	372. 89
	1.74	1.71	1.69	1.68	1.66	1.64
300	290. 35	307. 56	324. 57	341. 40	358. 06	374. 53
	1.73	1.71	1.69	1.67	1.66	1.64
400	292. 08	309. 27	326. 26	343. 07	359. 72	376. 17
	1.73	1.71	1.69	1.67	1.65	1.63
500	293. 81	310. 98	327. 95	344. 74	361. 37	377. 80
	1.73	1.71	1.69	1.67	1.65	1.63
600	295. 54	312. 69	329. 64	346. 41	363. 02	379. 43
	1.72	1.70	1.68	1.67	1.65	1.63
700	297. 26	314. 39	331. 32	348. 08	364. 67	381. 06
	1.72	1.70	1.68	1.67	1.65	1.63
800	298. 98	316. 09	333. 00	349. 75	366. 32	382. 69
	1.72	1.70	1.68	1.67	1.65	1.63
900	300. 70	317. 79	334 68	351. 42	367. 97	384. 32
	1.72	1.70	1.68	1.66	1.64	1.63
1000	302. 42	319. 49	336. 36	353. 08	369. 61	385. 95

PYROMETRIC INSTALLATIONS

FIG. 166.—*One of the high-temperature measurement laboratories at the Bureau of Standards*

In the foreground are placed some industrial types of optical and radiation pyrometers. The observer on the right is sighting into a "black body" furnace with the primary standard optical pyrometer

FIG. 167.—*One of the high-temperature measurement laboratories of the Bureau of Standards*

In the foreground are shown some industrial types of thermocouples and millivoltmeters. Furnaces for melting standard melting-point metals are shown in the background

FIG. 168.—*Six-point indicator with two temperature scales—one for rare-metal couples in high-speed steel furnaces and one for base-metal couples in carbon-steel furnaces*

Plant of a large Philadelphia tool company

FIG. 169.—*Two-point indicator in a furnace for heat treating airplane engine cylinders*

FIG. 170.—*Eighteen-point indicating pyrometer in rear of furnace of a malleable steel company*

FIG. 171.—*Measurements of furnace temperature with the optical pyrometer*

The operator is sighting with the disappearing-filament pyrometer into a furnace through a small observation hole in the door. True temperatures are obtained, since fairly good black-body conditions are assured (if the temperature distribution in the furnace is sufficiently uniform)

FIG. 172.—*Measurements of temperature of a tool with the optical pyrometer*

The operator is sighting with the disappearing-filament pyrometer at a small tool undergoing heat treatment. Since the temperature may be estimated to within 5° C, the most exacting specifications for heat treatment can be followed

FIG. 173.—*Measurements of ingot temperatures with the optical pyrometer*

The operator is sighting with the disappearing-filament pyrometer on an ingot which is passing from the furnace to the rolls. Although the observation is being made in the open, practically true temperature of the oxide surface is obtained on account of the very high emissivity of iron oxide. Small corrections for emissivity amounting to less than 10° C may be applied when necessary, as shown in Table 16 of the text

FIG. 174.—*Measurements of forging temperature with the optical pyrometer*

The operator is sighting upon an ingot during the forging process. In this manner the specifications for forging temperature of high-grade steel may be closely followed

FIG. 175.—*Use of pyrometry in the ceramic industry*

Rare-metal thermocouples are shown installed at intervals of 25 feet through the top of a tunnel kiln. The wiring is in conduit with outlets for connection to the thermocouple heads. Asbestos-covered compensated extension wire removes the cold junction to a point some distance from the kiln

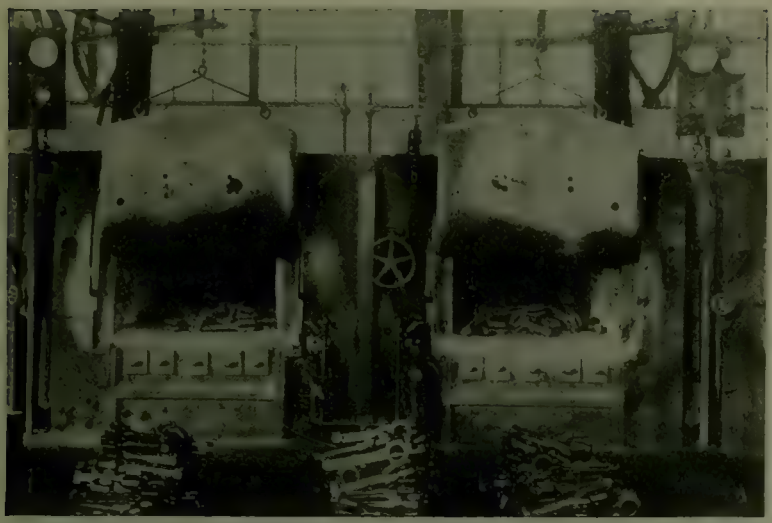


FIG. 176.—*Automatic signaling control*

In the upper corners of the illustration are shown indicators and colored electric lights mounted on panel boards. The signals, made by the red, white, and green lights, and by the auxiliary indicators are controlled by a recorder equipped for automatic signaling. The recorder is connected to thermocouples installed in the furnaces

FIG. 177.—*Furnace room of a steel plant where high grade automobile parts are heat treated*

Thermocouples are installed in each furnace, and a multiple-point indicator is shown mounted on a column in the center of the room

FIG. 178.—*Pyrometric recorders in a superintendent's office*

The thermocouples installed in the furnaces shown in Fig. 177 are connected to recorders in the superintendent's office. A continuous record is accordingly obtained of the operation of each furnace

FIG. 179.—*Central pyrometer station*

In a large plant it is frequently desirable to install all recorders in a single room. An attendant has charge of the installation and the maintenance of the records

FIG. 180.—*Central pyrometer station*

The recorders are equipped with devices for operating the relay switches at the left. By this means electric furnaces are maintained automatically at specified temperatures

FIG. 181.—*Automatic temperature control*

View of another installation of automatic control devices

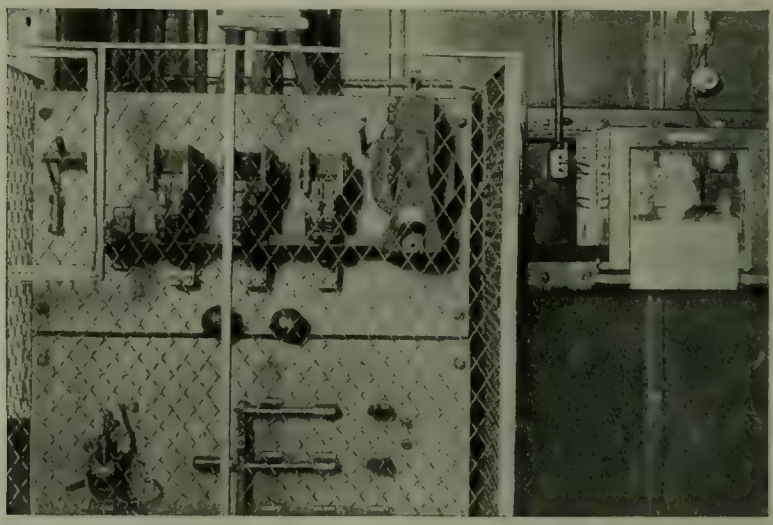


FIG. 182.—*Automatic temperature control*

The electromagnetic switch, operated by the relay, which in turn is operated by the recorder, is shown in greater detail

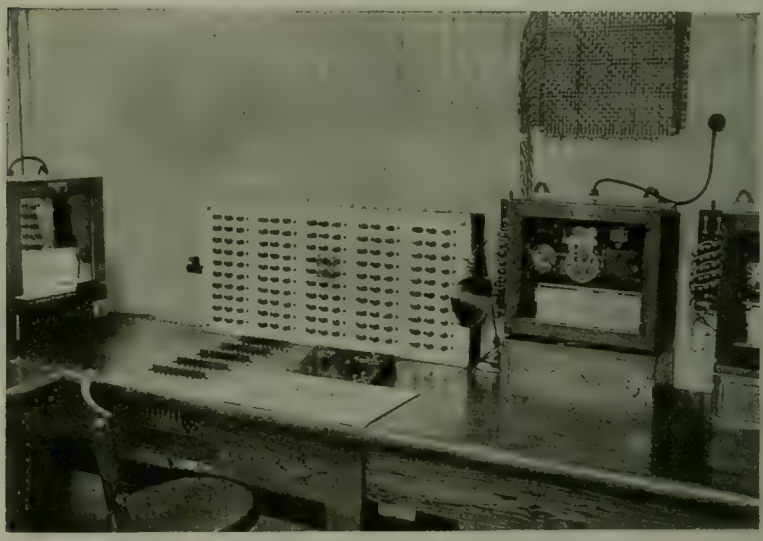


FIG. 183.—*Central pyrometer station*

The photograph shows one corner of a large central station. The plug board above is used for connecting the multiple-point recorders to any combination of furnaces. The switchboard is used for connecting couples to the potentiometer indicator and for transmitting signals to the furnace room

FIG. 184.—*The foreman's office in a pottery*

The foreman has in hand the complete record of the firing of a kiln. Close study of such records in correlation with the quality of the ware produced is doing much to replace art by science in the ceramic industries

FIG. 185.—*Heat treating department of a large plant*

Each one of the many furnaces is equipped with an indicator and with colored signaling lamps. Such control results in the production of a uniform and high grade product

INDEX

	Page
Absorption devices	269–279
absorption-glass standardization	271
absorption-glass transmission	269
red-glass standardization	272
sectored-disk standardization	271
Acheson graphite (*see also* Graphite)	237
Alarm, temperature	167, 169
Alloys, melting points	192, 197, 210
Alumel. *See* Chromel.	
Alumina (*see also* Crucibles)	10, 212, 215, 218
Aluminum, certified samples	236
graphite sheath	194, 238
melting point	10, 192, 144, 235, 236, 268
Alundum, cement in furnace construction	195, 206, 240
melting point	225
protecting tubes	93
Antimony, melting point	9, 10, 192, 194, 209, 235
Applications of pyrometry, industrial	282–299
cement kilns	287
ceramic industries	289
coke industry	293
glass manufacture	283
steel manufacture (*see also* Steel manufacture)	296
Arsem furnace	213, 218, 221, 268
Atmospheres, proper, for molten materials	194, 212
Automatic, alarm	167, 169
signaling device	170
temperature control. *See* Temperature control.	
Barkley, Kreisinger and	249
Benzophenone, boiling point	10, 225
Bimetallic, spring compensator	55
thermometer	11
Black body	110, 126, 259, 267, 269, 274
Boiling points	9, 225, 278
Boltzmann. *See* Stefan.	
Borax, melting point	204, 206
Bristol, automatic signaling device	171
Bronze, melting range	201
Brooks	249
Brown and Murray	292
Brown, automatic temperature control	176
improved heatmeter	39, 248, 256
potentiometer	46
precision heatmeter	47
radiation pyrometer	122
recorder	142–144
transformation-point recorder	162
Burgess, G. K	162, 200, 210, 215, 216, 237, 273, 287, 298
Burgess, H. A	278, 282
Cadmium, melting point	10, 192, 194
Callendar, resistance-thermometer	129
equation	209
Calorized iron protecting tubes	94
Carborundum protecting tubes	91
Cement, alundum, melting point	225
Cement kilns	287
Ceramic industries	289
Ceramic materials, melting points	218
Chlorides, melting points	203
Chromel	113
alumel thermocouples	21, 286, 287–293
furnace heater	195, 242, 243, 306, 307
protecting tubes	92, 287
Chromium, melting point	212
oxide, melting point	218
Chromon protecting tubes	94
Clay (*see also* Fire clay)	95, 225, 294
Cleveland deflection potentiometer	53
recorder	142
Coal ash	223
Cobalt, melting point	212
Coke industry	293
Cold junction (*see also* Thermocouple)	21, 24
buried	57, 76, 87
compensation, bimetallic spring	55
compensating leads	56, 76, 77, 83
potentiometric	58
shunting method	60
Wheatstone bridge	63
compensator	249, 255
corrections	54, 55, 246
ice box	233
thermostated	56
water-jacketed	56, 227
Commutating switch (*see also* Switchboard)	82
Comparator, thermometer testing	225
Compensating leads or cold-junction extension leads	56, 76, 77, 83
Constantan, in thermocouples	21, 280, 306, 307
Conversion tables. *See* Tables.	
Copper, certified samples	236
emissivity	114, 127
eutectics	10, 198, 267
melting point	9, 192, 194, 197, 209, 234, 268
oxide	127, 197, 209
Crowe	211
Crucibles, acheson graphite	193, 237, 267
alumina	193, 212
Dixon graphite	207, 237
duriron	244
fire clay	203
for black body	267
for molten palladium	213
magnesia	193, 212
magnesium aluminate	193, 212
nickel	203
platinum and platinum alloys	203, 205
porcelain	193

Page

Curves, cooling and heating 234
derived differential 164
freezing 196
inverse-rate 162
obliquity 189, 203
time-temperature 161

Dana 187
Deflection potentiometer 42, 48, 262
Depth, immersion of thermocouples. *See* Thermocouples.
Dickinson 226
Diesselhorst potentiometer 233
Differential galvanometer 130
Diopside, melting point 204, 206
Disappearing filament optical pyrometer 105, 210, 223, 260
Dixon. *See* Crucibles.
Duriron 94, 244

Effective wave length 270
Electromotive force of thermocouples ... 20, 226–236
Emissive powers 95, 114
Englehard, platinum, platinum-rhodium thermocouple 64, 306, 309
recorder 145
Eutectic 10, 199, 268
Extension leads. *See* Compensating leads.

Fairchild 272, 273, 289
Ferguson 219
Fery pyrometer, optical 98, 266
radiation 122, 274
Field 223, 292
Fieldner 223, 292
Fire bricks and clays, melting points ... 218, 220, 225
Fixed points (*see also* Melting points; Boiling points) 9
F. and F. optical pyrometer 104, 266
Foote 10, 39, 60, 211, 256, 270, 272, 273, 274, 298
Forsythe 216, 273
Foster radiation pyrometer 121
Freeman 200
Freezing point. *See* Melting points.
Furnaces, electric, Arsem 213, 218, 221, 268
carbon resistance 221
chromel 195, 241, 243
for critical points 200
for melting points, metals 195, 208
refractories 221
salts and silicates 205, 274
for pyrometer standardization, optical .. 213, 258, 268, 269
thermocouples 237, 240, 241
iridium tube 215
Northrup-Ajax 215
gas for melting points 208, 222, 224
for thermocouple standardization 237, 242
control. *See* Temperature control.
Fusible cones 222, 289, 292

Galvanometer, differential 130
for radiation pyrometer 277
for thermocouples 26–43, 138–156
standardization 248, 253, 257

Page

Gas, furnaces for melting points (*see also* Furnaces) 208
ideal 8
pyrometer 11, 18, 135
thermometer 8, 9, 11, 18, 135
Gauze, glowing, temperature 114–116
Geophysical laboratory 193, 204
Glass, absorption and transmission 270
annealing 285
manufacture, pyrometry applied 283–287
Gold, melting point 192, 194, 208, 209, 268
Graphite 93
Acheson 193, 194, 207, 237, 238
black body 267, 274
Dixon 207, 237

Hall 223, 292
Harper 210
Harrison 39
Harrison-Foote compensated galvanometer .. 39, 248, 256
Heatmeter 39, 47, 248, 256
High temperature thermometry (*see also* Thermometry) 10–20, 225
Homogeneity test 228, 245
Hoskins recorder 142, 145
Hot junction. *See* Thermocouples.

Ice box for thermocouple junctions 233
Ice point 9, 278
Ideal gas 8
Immersion of thermometers 15, 226
Impervite 194
Indicator. *See* Measuring instruments.
Ingersoll, Mendenhall and 216
Insulating tubes for thermocouples. *See* Protecting tubes.
Installations, illustrations of pyrometric ... 310–319
Iridium 22, 203
tube furnace 215
Iron 113, 127
melting point 212
oxide 113, 127
protecting tubes 94
thermocouples 21, 286, 306

Jaeger 206
Johnson-Matthey thermocouples 64, 306
Junction box 83–87

Kanolt 211, 213, 218, 219, 225
Kreisinger and Barkley 299

Lamps, electric, calibration of optical pyrometers 260
melting-point determination 216
optical pyrometer 260
Langmuir 216
Le Chatelier 237
thermocouples 21, 55, 64, 306, 308
Lead, certified samples 236
freezing point 10, 194
Leads, compensating 56, 76, 77, 84
thermocouples 21
resistance 28–33
correction 33, 39, 41

Page

Leakage, thermoelectric circuit 205
Leeds and Northrup, automatic signaling recorder 171
automatic temperature control 177
cold-junction compensator 58, 253
deflection potentiometer 252
recorder 156, 253
transformation-point indicator 163
Lime, melting point 218
Liquid-filled thermometers 11, 18, 135
Luminosity 270
Lummer-Kurlbaum black body 267

Magnesia, melting point 218
Magnesium aluminate (*see also* Crucible) .. 193, 212
Measuring instruments, thermoelectric
calibrated with thermocouples 257
calibration 248, 253, 263
deflection potentiometers 43, 52, 262
galvanometers ... 26, 53, 138, 156, 248, 253, 257, 277
heatmeter 39, 248, 256
indicators 21, 26, 75, 78, 134, 161–165
multipyrograph 148
potentiometers. *See* Potentiometers.
pyrovolter 248
range control board 51, 54
recorders (*see also* Galvanometers; Potentiometers) 75, 134–164
tapalog 153
use of, with calibrated thermocouples. 248–253
Melting point, methods at high temperatures 187–225
by micropyrometer 216
by miscellaneous methods 207, 213, 216
by optical pyrometer 210–217
by resistance thermometer 209
by thermocouple 189–209
effect of pressure 188
for optical pyrometer standardization .. 268
for thermocouple standardization 192–197, 200, 206, 234–236
furnaces, electric 195, 206, 208, 213, 221, 237
gas 208, 222, 223, 237, 242
heating or cooling rate 213, 221
obliquity of curve 189, 196, 199, 202
of alloys 197, 198, 210
of alundum cements 225
of ceramic materials 218–223
of chlorides 203
of coal ash 223
of clays 225
of copper oxide 127, 197, 209
of eutectics 10, 198, 199, 268
of fire bricks 212–223, 225
of metals 9, 10, 192–202, 212, 234–236, 268
of nitrates 203
of refractories (*see also* Refractories) 219–225
of salts 206
of sands 225
of silicates 203, 206
of sulphates 203
standard samples 236
surfusion or undercooling 235
wire method 207
Melting-range determination 200
Mendenhall and Ingersoll 216

Page

Metals, for thermocouples 21
melting points. *See* Melting points.
molten-bath 243
surface temperature 298
Mercurial thermometer 10–20
accuracy 12
filling under pressure 16
graduation 12
industrial 12
Mesa 201
Mertelmeyer 60
Merwin, Ferguson and 219
Meyer, Pirani and 216
Millivoltmeters. *See* Galvanometer; Measuring instruments.
Micropyrometer 216
Mohler 272, 273
Molybdenum in thermocouples 22
Mueller 10, 200, 278, 279, 282, 299
Murray, Brown and 292

Naphthalene, boiling point 10, 225
Nela Research Laboratory 260
Nichrome 21, 92, 113, 243, 287
Nickel 94, 127, 203, 260
melting point 10, 194, 212
oxide 113, 127, 275
resistance thermometer 281
strip 275
Nitrates, melting points 203
Nitrogen thermometer 8
Northrup-Ajax furnace 215

Optical pyrometers 94, 98–118, 258–273
absorption, devices 263, 269
window 117
applications 108–110, 210, 213, 284–299
black body and nonblack body 110–116
disappearing-filament type 105–108, 210, 223, 260, 267, 292
F. and F 104, 266
Fery 98, 266
glowing gauze 114–116
micropyrometer 216
Scimatco 103
sectored disk 271
Shore pyroscope 99
sighted into closed tube 118
standardization 95, 97, 258–273
temperature-scale 96
extension 263
theory 94
Wanner 100–103, 263–266
Orton 289

Palladium, melting point 10, 208, 212
Parasitic emfs. *See* Homogeneity.
Peltier emf 20
Pirani and Meyer 216
Planck, Wien-, radiation law 96
Platinum, crucibles 202–205
melting point 10, 212
resistance thermometer. *See* Resistance thermometer.
thermocouples. *See* Thermocouples.
Porcelain, crucibles 194
protecting tubes 91, 193, 227, 238, 294

Page

Potentiometers ... 43–46
deflection ... 43, 48–54, 202
precision ... 252–253
shielding ... 205
standardization ... 253–258
used with optical pyrometers ... 262
used with standardized couples ... 249–252
Potentiometric, cold-junction compensator ... 58, 249–252, 255, 257
recorder ... 156–161, 177, 255
Protecting tubes for thermocouples ... 24, 26, 78, 89–94
alundum ... 93
calorized iron ... 94
carborundum ... 91
characteristics ... 89
chromel ... 92, 287
chromon ... 94
clay ... 92, 294
cleaning ... 248
corundite ... 92
duriron ... 94
fused quartz or silica ... 90, 193
graphite ... 93, 194, 238
impervite ... 194, 294
in salts and silicates ... 202, 205
iron ... 94
nichrome ... 92, 287
nickel ... 94
porcelain ... 91, 193, 238, 294
precautions in use ... 238
Pyrex glass ... 193
requirements ... 89
sillimanite ... 194
steel ... 94
usalite ... 194, 294
Pyrometry, industrial applications ... 282, 299
Pyrovolter ... 47, 248

Quartz, melting point ... 204, 219
thermocouple protecting tubes ... 90, 193

Radiation, intensity ... 95
laws ... 9, 96, 97, 118, 270
Radiation pyrometer ... 94, 97, 118–126, 273–277
advantages and disadvantages ... 125
applications ... 290, 293, 298
black-body conditions ... 126–127, 274
emf measurement ... 277
error sources ... 125, 274
Fery ... 122, 274
Foster ... 121
recorder ... 135
standardization ... 95, 273–277
temperature scale ... 97
extension ... 276
theory ... 194
Thwing ... 119
Range control board ... 54
Raoult-Van't Hoff ... 197
Rawdon ... 298
Recording pyrometers ... 17, 134–164
automatic signaling ... 170
Bristol ... 143
Brown ... 142, 144, 162
circular chart ... 142
Cleveland ... 142

Recording pyrometers—Continued Page
damping vibrations ... 75
drum chart ... 144
Engelhard ... 145
forms of records ... 135
galvanometric ... 138–156
gas ... 135
Hoskins ... 142, 145
industrial ... 181–187, 284–289
Leeds and Northrup ... 163, 156–161, 171
liquid ... 135
motive power ... 140
multiple point ... 138, 168
multipyrograph ... 148
parallel connections with indicators ... 79
potentiometric (*see also* Potentiometers) 156–161
radiation ... 135
resistance ... 135
roll chart ... 144
saturated vapor ... 135
Siemens and Halske ... 139
standardization ... 255
Stupakoff ... 144
tapalog ... 153
Taylor ... 147
Thwing ... 152
transformation point ... 161–165
Wilson-Maeulen ... 153
Refractories, melting points (*see also* Silicates) ... 206, 217–225
table ... 225
Resistance pyrometers ... 8, 9, 11, 127–134
agreement, with thermodynamic scale ... 9
with thermocouple ... 209
as standard pyrometer ... 209
compensating lead wire ... 129
deflection galvanometer ... 132
for melting points ... 209
formula ... 129, 209, 281
four-lead ... 132
industrial ... 128
leads ... 282
measuring instrument ... 129
nickel coil ... 281
operating principle ... 127
platinum ... 9, 209
potential terminals ... 130
standardization ... 278–282
temperature scale ... 9, 209
with indicator ... 281
Rosenhain ... 200, 202
Rhodium. *See* Thermocouples.
Ruthenium, in thermocouples ... 22

Salts, furnaces for melting points ... 206
melting points ... 204, 206
Sands, melting points ... 225
Scimatco optical pyrometer ... 103
Scott and Freeman ... 200
Sectored disk ... 271
Seebeck ... 20
Seger cones ... 222, 289, 292
Shielding thermoelectric circuits ... 205
Shore pyroscope ... 99
Siemens and Halske ... 139
Signaling ... 167, 169
Silicates, furnace for melting points ... 206
melting points ... 191, 202–206

Page

Silicon, melting point ... 212
Sillimanite ... 194, 211, 222
Silver, melting point ... 10, 192, 194, 198, 209, 268
  effect of absorbed oxygen ... 198
Sodium chloride, for checking pyrometers ... 207, 244
Sosman ... 222, 289
Standard cells, standardization ... 253
Standard melting point samples ... 236
Standardization of pyrometers ... 225–282
  optical ... 95, 258–273
  radiation ... 95, 97, 273–277
  resistance ... 278–282
  thermoelectric ... 192, 194, 200, 206, 207, 226–247
  thermometric ... 225
Steam point ... 8, 9, 225, 278
Steel manufacture, pyrometry ... 296–299
Stefan-Boltzmann radiation law ... 9, 97, 118, 217
Stem correction to mercurial thermometers ... 15, 226
Storage cells, for potentiometers ... 233
Stupakoff recorder ... 144
Sulphates, melting points ... 203
Sulphur boiling point ... 9, 10, 225, 278
Switches, commutating ... 82

Tables, annealing temperatures of optical glasses ... 286
  conversion centigrade and Fahrenheit ... 301, 302
  cold-junction correction factors ... 55
  emissive powers ... 114
  logarithms of tangents ... 304
  melting points, metals ... 10, 194, 212
  refractories ... 206, 218, 225
  salts ... 286
  silicates ... 206
  softening temperatures of fusible cones ... 292
  thermoelectric ... 306, 307
  true temperatures verses apparent temperatures ... 113, 114, 127
Tapalog ... 153
Taylor recorder ... 147
Temperature coefficient of galvanometers ... 29, 41
Temperature control ... 96, 134, 165–187
  automatic ... 174
  factors increasing difficulty ... 165
  glass manufacture ... 283
  location of pyrometer in furnace ... 166
  necessity of pyrometers ... 165
  signaling devices ... 167, 169
Temperature scales ... 8
  optical pyrometer ... 96
  radiation pyrometer ... 9, 97
  resistance pyrometer ... 9, 209
  thermodynamic ... 8
  thermoelectric pyrometer ... 9, 209, 236
Test set ... 33
Thermocouples, base-metal ... 24
  chromel-alumel ... 21, 286, 287, 293, 295, 306
  compensating leads ... 76
  copper-constantan ... 21, 306
  iron-constantan ... 21, 286, 306
  method of making junctions ... 247
  nichrome-constantan ... 21
  standardization ... 242–246
  use ... 286, 287, 291, 296
  calibration tables ... 306–308

Thermocouples—Continued Page
  cold junctions ... 21, 24
    buried ... 56, 76
    compensation, bimetallic spring ... 55
      compensating leads ... 56–58, 76, 77, 84
      potentiometric ... 58–60, 248
      shunt ... 60
      Wheatstone bridge ... 63
    correction factor ... 54, 55, 246
    ice box ... 233
    junction box ... 83–87
    mounted couples ... 227
    temperature ... 54–64, 87, 241
    thermostated ... 56
  depth of immersion ... 88, 196, 229
  fused junctions ... 25, 247
  homogeneity ... 22, 227
  hot junction ... 21, 25, 240, 247
  installations ... 74–94
    common return ... 76, 78
    damping vibrations ... 75
    location, couples in furnaces ... 293, 295
    precautions ... 74
    weatherproof head ... 75
    wiring ... 74
    wiring diagrams ... 77–83
  insulation (*see also* Protecting tubes) ... 24
  iridium ... 22
  Le Chatelier ... 21, 55, 64, 306, 308
  lead wires (*see also* Compensating leads) ... 21
  location, furnace ... 75, 81, 166
  measuring instruments used ... 248–258
  melting point, determinations ... 189–209
  metals used ... 21
  mounted ... 24, 227
  platinum, platinum-rhodium ... 22, 55, 64, 226, 285, 290, 291, 293
    agreement, with resistance pyrometer ... 209
      with thermodynamic scale ... 9
    annealing ... 227
    calibration data ... 306–308
    contaminated ... 229
    for precision work ... 192
    for primary standards ... 239
    homogeneity test ... 228–232
    protection ... 238
    standardization ... 192, 194, 200, 206, 207, 227–242
  Peltier emf ... 20
  protecting tubes. *See* Protecting tubes.
  reproducibility ... 23, 64–74
  resistance of leads and errors due to ... 28–33
    correction ... 39
    measurements ... 33
  seat of emf ... 20, 227, 245
  size of wires ... 22, 24
  standardization ... 227–246
    for industrial laboratories ... 227, 236, 240
    furnaces ... 237, 240, 243
    with measuring instrument ... 254
  Thomson emf ... 20
Thermoelectric circuits, leakage, and shielding ... 205
  emfs ... 20, 227, 228
  measuring instruments. *See* Measuring instruments.
  recorder. *See* Recorder.

	Page
Thermometers, high-temperature	10–20
accuracy of high-grade	12
annealing	17, 226
bimetallic	11
classification	11
filling under pressure	16
gas	8, 9
gas pressure	11, 18, 135
graduation	12
graphite-metal expansion	11
indicating	17–20
industrial	12
liquid pressure	11, 18, 135
liquids used	18
mercurial	11
pressure, gas, liquid, vapor	11, 17, 135
resistance. *See* Resistance pyrometry.	
standardization	225
stem correction	15, 226
vapor pressure	11, 17, 135
Thomson, bridge	130
emf	20
Thwing, radiation pyrometer	119
recorder	152
Tin, bath	243
certified samples	236
melting point	192, 194, 236
Tool	286
Transformation-point indicators and recorders	161–165
Transmission of absorption glass	220
Tungsten (*see also* Lamps)	22
melting point	10
Undercooling	192, 235
Usalite	194, 294
Valasek	286
Van't Hoff, Raoult-	197
Vapor-pressure thermometer	11, 17, 135
Veritas firing rings	289
Visibility	270
Waidner	9, 210, 215, 216, 282, 298
Waltenberg	298
Wanner optical pyrometer	100–103, 263–266
Wartenberg	216
Wave length, effective	270
Weatherproof thermocouple head	75
Wheatstone bridge	35, 130, 131, 282
White	189, 205
potentiometer	233
Wien-Planck radiation law	9, 96, 271
Wilhelm	10, 226
Wilson-Maeulen recorder	153
zone box	86
Wiring diagrams	78–83
Wiring for thermocouples	74
Worthing	216, 273
Zinc, freezing point	9, 10, 192, 194, 197, 236
certified samples	236
Zone box	86

DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 171

AUTOMATIC APPARATUS FOR INTERMITTENT TESTING

BY

G. W. VINAL, Associate Physicist

L. M. RITCHIE, Assistant Chemist

Bureau of Standards

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FIG. 1.—*Complete view of the new automatic apparatus for intermittent testing. The clock controls the selective relays under the glass case on the table. This apparatus can carry on simultaneously a number of different tests on dry cells and storage batteries. Dry-cell tests are made in the constant-temperature room at the left. The voltage of each group of batteries may be read at the telephone jacks beneath the window*

AUTOMATIC APPARATUS FOR INTERMITTENT TESTING

By G. W. Vinal and L. M. Ritchie

1. INTRODUCTION AND APPLICATION

This apparatus has been devised to meet the needs of the Bureau of Standards in making tests of dry cells and storage batteries, but is applicable to nearly any form of intermittent test requiring the closing of electrical circuits at regular time intervals.

The usual tests of dry cells which are made at the present time are arbitrary. They involve the discharge of the cells through resistances for varying periods of time, depending upon the service for which the particular cells are designed. The telephone test, frequently referred to as the "A. T. and T. test," consists of a discharge of 3 cells in series during 4 minutes per hour for 10 consecutive hours a day, but omitting every other discharge period on 1 day of the week. The "ignition test" for cells which are designed for a heavier type of service involves a discharge of the group of cells for 1-hour periods twice each day. The "flash-light test" requires a discharge of the cells for 5 minutes once during 24 hours. The details of these tests may be found in Circular No. 79, Electrical Characteristics and Testing of Dry Cells, published by the Bureau of Standards.

Additional tests which may be made simultaneously with these include the life-testing of storage batteries and intermittent tests of caustic-soda primary batteries, such as are used for railway signaling. The life tests of storage batteries consist of a continuous series of charges and discharges, 2 complete cycles being made in each 24 hours, without requiring the presence of an observer. Researches on the capacity of various batteries for differing periods of discharge are planned also.

2. DESCRIPTION OF CONTROL APPARATUS

A general view of the apparatus[1] is shown in Fig. 1. The pendulum clock, in which an electrical contact has been placed, closes the circuit once every minute and furnishes an electrical

[1] We are indebted to Matthew Harrison and F. M. Defandorf for valuable assistance.

impulse to one of two selective relays which are contained in the glass case on the table. The details of these relays may be more clearly seen in Figs. 2 and 3. As each impulse is received, the shaft at the left-hand end is advanced by one-sixtieth of a revolution, and therefore it makes 1 complete revolution per hour. Once an hour this shaft furnishes a similar impulse to the coils at the other end of the apparatus, which causes the shaft at the right-hand end to rotate. There are 24 teeth in the wheel at this end, and this shaft therefore makes 1 revolution in 24 hours. The third shaft is geared to the 24-hour shaft in the ratio of 1 to 7 and makes 1 revolution in a week. By placing suitably-designed commutators on these three shafts the apparatus may be made to control any required periodic test.

Owing to the inertia of the commutators on these shafts, it was necessary to use a driving mechanism similar to the escapement of a clock, in order that the shaft might not turn by more than the given angular amount when the armature is operated by the current in the magnet coils. The commutators are made of hard rubber, with metallic segments having platinum surfaces. The brushes which bear upon the commutators are of phosphor-bronze, with contact points of platinum-iridium. In the illustration, Fig. 3, the commutators for making the telephone test may be seen. The commutator on the hour shaft closes the circuit for 4 minutes. The commutator on the day shaft has 10 segments which are staggered, every other one of these being made inoperative one day a week by the insulated portion of the commutator on the week shaft. Commutators for the other tests which are being controlled by this apparatus are not shown in this picture.

When the circuit through the contacts on the commutators is complete, the current closes one of the master relays which may be seen in Fig. 1 above the automatic apparatus. These are telephone relays of the type No. 118, Western Electric, to which small phosphor-bronze springs have been added to prevent sticking of the relay owing to the current of the multiple-switch solenoid, which flows through the contact for a considerable time. These springs are operative only at the instant when the relay begins to release the armature and do not affect the relay when the magnetized coil first acts on the armature after the circuit is closed. One master relay is provided for each different test excepting for the storage-battery life-test circuit, for which one relay operates the charging circuit and a second relay the discharge circuit. Each relay is connected with an electric counter (Kellogg

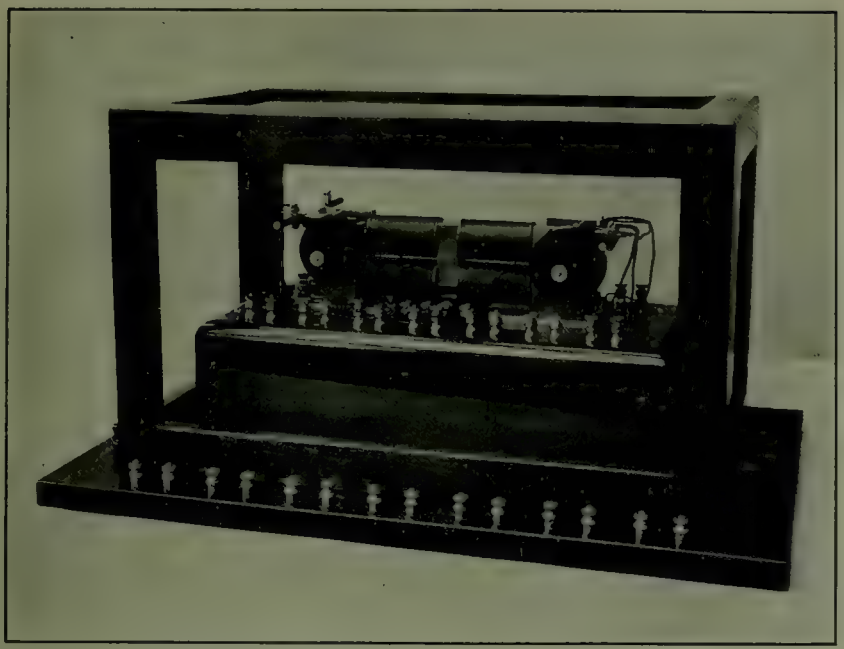

FIG. 2.—*The control apparatus. The shaft at the left makes one revolution per hour, the shaft at the right one revolution per day, and a third shaft at the back (see Fig. 3) one revolution per week*

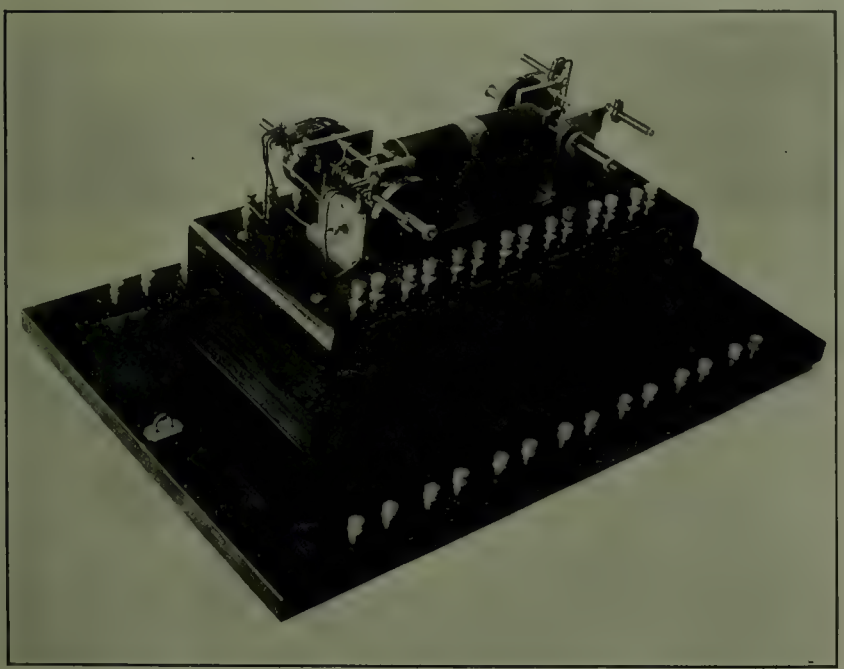

FIG. 3.—*Detail view of the apparatus. The driving mechanism is like the escapement of a clock. Segments on the commutators have platinum surfaces. The brushes are of phosphor-bronze with platinum-iridium contact points. Only a few of the commutators are shown*

message register), which records the number of times that the circuit is closed. A secondary contact in each message register is utilized to light a small signal lamp to indicate which circuit is in operation.

The diagram of the circuits is shown in Fig. 4. Between the clock and the control apparatus there are two relays. The first is within the clock itself; the second is in the box which may be seen immediately beneath the clock in Fig. 1. This second relay is a matter of convenience for other purposes, but is not necessary for the operation of the apparatus. In the locked box with this

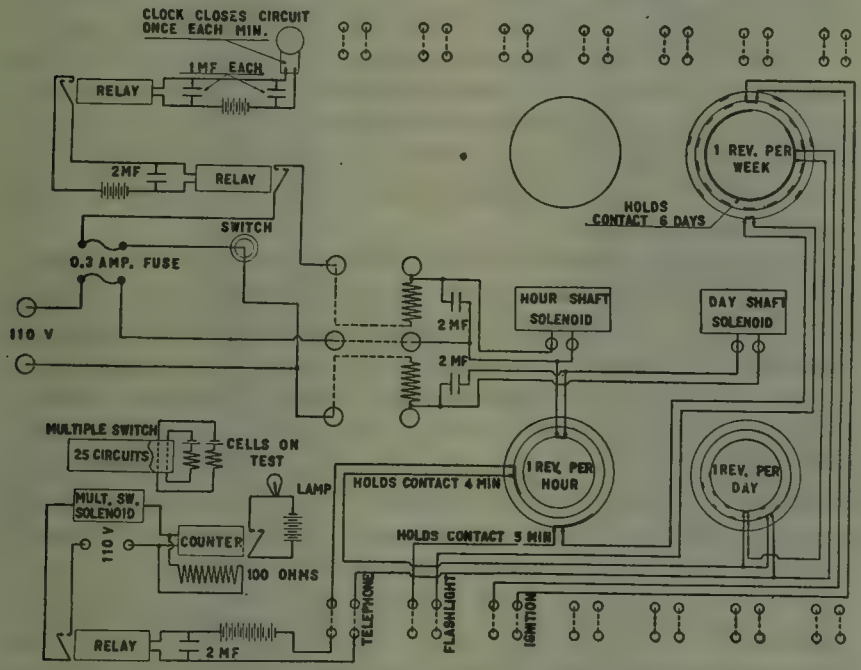

FIG. 4.—*Diagram of the circuits*

relay is a snap switch for the 110-volt circuit, also 2 fuses made of gold foil which will burn out if the current exceeds 0.3 ampere. They are to protect the solenoids of the automatic apparatus.

The batteries indicated in Fig. 4 may be seen beneath the table in Fig. 1. They are of the copper oxide-zinc type, approximately 300 ampere-hour capacity, made by the Edison Primary Battery Co., the National Carbon Co., and the Waterbury Battery Co. They are assembled in a box on casters and have flexible electrical connections so that they may be rolled out into the room for examination or voltage measurements whenever required.

3. MULTIPLE SWITCHES AND TEST BOARDS

For the dry-cell testing it is necessary to close a large number of independent circuits simultaneously, For this purpose multiple switches, as shown in Fig. 5, are used. The contacts are mercury cups with double break. The switch shown is constructed of "Formica." The upper part of the switch is mounted on pivot hinges and is operated by the solenoid. The current required to operate this solenoid is 0.09 ampere, which is sufficient to give the switch a positive action without being too much current for the secondary contact of the relay. The counterweight, which may be seen at the back, is adjusted to open the switch by gravity and keep it open except when current is flowing through the solenoid. In case of failure of the power circuit at any time the switch opens and avoids overdischarge of the batteries. By disconnecting the switch from the solenoid the top of the switch can easily be thrown back for an examination of all the mercury contacts.[2]

Dry cells such as are in tubular flash-light batteries are easily tested by mounting them on the board shown in Fig. 6. Sliding contacts at the lower part of the board permit an adjustment for any length of battery. A screw with a blunt end supports the battery at the bottom (zinc electrode) and a similar screw with a sharp point at the upper end of the battery makes contact with the brass cap on the carbon rod, clamps the cells together, and securely holds the battery in position. The discharge resistances are 4 ohms for each cell in the battery. These resistances have, therefore, been made up as unit coils of the same dimensions having 4, 8, and 12 ohms. Each coil has lead terminals at its ends, which are gripped by the pointed screws that hold the coil in place—above the battery which is on test. Potential leads from the terminals of each battery pass in a cable along the back of the board and are brought to the telephone jacks beneath the window of the constant-temperature room shown in Fig. 1. Readings are rapidly taken on a voltmeter by the telephone-plug connection while the batteries are discharging.

4. SUMMARY

The apparatus which has been described was designed primarily for testing dry cells and storage batteries. It automatically closes the contacts for the discharge of the dry cells at predetermined

[2] Since this paper was written we have also used 31-contact relays (Type 331H) made by the North Electric Co. These are provided with wiping contacts suitable for closing the battery circuits.

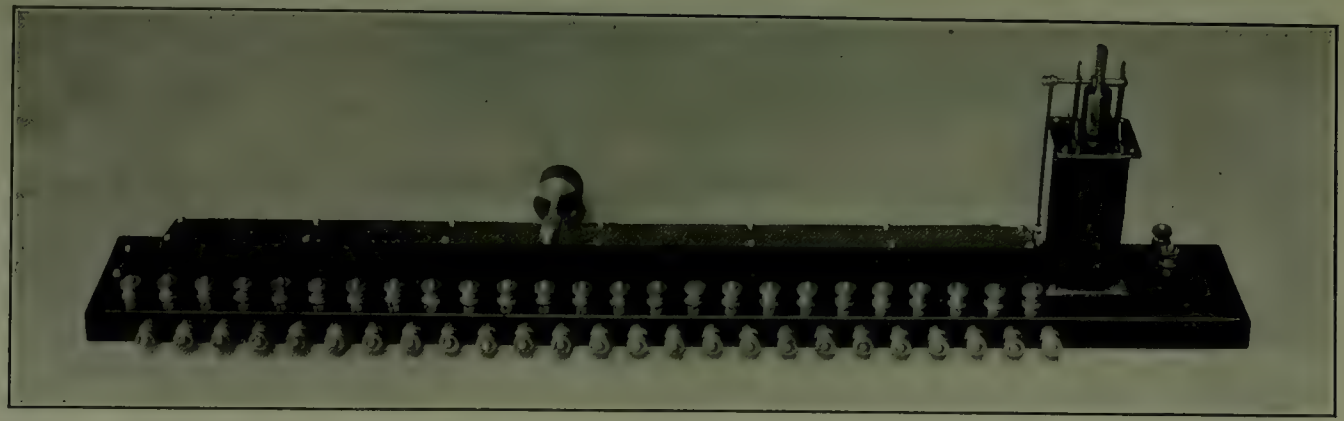

FIG. 5.—*Multiple switch used for dry-cell testing. It is operated by the solenoid at the right, which requires 0.09 ampere. The switch closes 25 circuits for the test board shown in Fig. 6. It is opened by gravity*

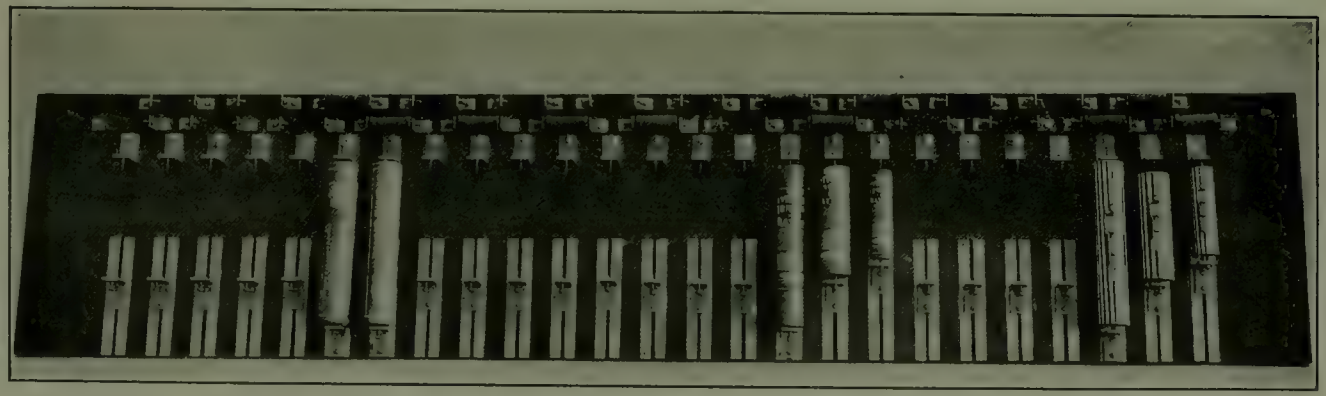

FIG. 6.—*Test board for flashlight batteries. Adjustable contacts provide for any length of battery. Resistance units for the discharge are mounted at the top. Potential leads for each battery are brought to the telephone jacks beneath the window in Fig. 1*

times and controls both the charge and discharge of storage batteries on life tests. The apparatus consists of 2 selective relays having 3 rotating shafts which make complete revolutions in 1 hour, 1 day, and 1 week, respectively. The coils of the first relay are energized once every minute by the clock shown in Fig. 1. The coils of the second relay are energized once each hour by the first relay. In this way rapidly moving parts are eliminated and the accuracy of the time intervals increased. Any periodic test may be controlled by this apparatus by means of suitably designed commutators which are placed on the rotating shafts. The contacts on the commutators are platinum against platinum iridium. Test boards for various sizes of dry cells with multiple switches to close the individual circuits are described.

WASHINGTON, April 22, 1920.

DEPARTMENT OF COMMERCE

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OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

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CAST IRON FOR LOCOMOTIVE-CYLINDER PARTS

BY

C. H. STRAND, Associate Physicist

Bureau of Standards

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By C. H. Strand

CONTENTS

	Page
Part 1. Investigation—Methods and results	3
I. Introduction	3
II. Previous work	6
1. Review of literature	7
2. Data furnished by U. S. Railroad Administration	8
(*a*) Foundry practice	8
(*b*) Tests made by U. S. Railroad Administration	9
III. Investigation at Bureau of Standards	10
1. Mechanical tests	10
(*a*) Transverse tests	10
Arbitration bars	10
Packing rings	11
(*b*) Tension tests	12
Arbitration bars	12
Packing rings	13
(*c*) Brinell hardness tests	14
Arbitration bars	14
Packing rings, bushings, and similar parts	15
(*d*) Chill tests	15
2. Chemical composition	16
(*a*) Arbitration bars	16
(*b*) Packing rings, bushings, and similar parts	17
3. Metallographic examination	18
IV. Comparison of properties of air-furnace and cupola cast iron	20
V. Conclusions and recommendations	20
Part 2. Proposed specification for cast-iron cylinder parts for locomotives	22

PART 1

INVESTIGATION—METHODS AND RESULTS

I. INTRODUCTION

Frequent renewal of cylinder parts of locomotives results in greatly increased cost of maintenance to the railroads, and consequently the quality of the cast iron entering into their construction is a matter of paramount importance, particularly from the standpoint of wear. These parts include piston-valve bushings, piston-valve packing rings, piston-valve bull rings, cylinder bushings, piston packing rings, and piston-head or bull rings.

It was found that ordinary high-silicon cast iron gave unsatisfactory wear, particularly in modern superheater locomotives, and the tendency has been toward a harder and stronger iron.

At the request of the former U. S. Railroad Administration the Bureau of Standards has investigated the mechanical, chemical, and microscopical properties of a number of packing rings furnished with service-mileage records, as well as arbitration-test bars, chill-test specimens, and miscellaneous samples from different manufacturers. All of this material was cast iron such as used for the various cylinder parts. It was desired at the same time to review the previous work and specifications on this subject, to ascertain as far as possible the practices of the different foundries and to suggest such revision of existing specifications as would be warranted by the results of the present and of earlier investigations.

The samples, 131 in number, were furnished to the Bureau of Standards by the U. S. Railroad Administration. The first samples were received April 2, 1919, and the last September 26, 1919; they were numbered 1 to 131, inclusive, in order of receipt, and their identification marks and history are to be found in Table 1. In accordance with the policy of the Bureau of Standards, the names of the manufacturers are withheld. In the course of this paper the nine foundries are designated by the letters A to I, inclusive.

TABLE 1.—Identification and History of Material Received for Examination

No.	Manufacturer	Service record	Description
		Miles	
1-26, inclusive	B	93 000	26 pieces constituting a packing ring removed for purpose of this investigation after giving remarkably long service.
27	C	Never in service	A small piece of packing ring of iron claimed to give excellent results.
28	A	do	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Sept. 4, 1918.
29	A	do	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Sept. 10, 1918.
30	A	do	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Oct. 16, 1918.

TABLE 1—Continued

No.	Manufacturer	Service record	Description
		Miles	
31	A	Never in service	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Jan. 17, 1919.
32	A	do	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Feb. 4, 1919.
33	A	do	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Feb. 15, 1919.
34	A	do	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Feb. 20, 1919.
35	A	do	Broken arbitration-test bar representing packing ring and cylinder iron furnished for U. S. Railroad Administration engines. Tests were made in presence of U. S. Railroad Administration inspector, Mar. 10, 1919.
36	A	do	Piece of cylinder packing ring, stamped contract B 1492, for use on U. S. Railroad Administration engines.
37	A	do	Piece of cylinder bushing blank, stamped contract B 1500, for use on U. S. Railroad Administration engines.
38	A	do	Piece of cylinder bushing blank, order 201, for use on U. S. Railroad Administration engines.
39	A	do	Piece of valve-chamber bushing blank, order 201, for use on U. S. Railroad Administration engines.
40	A	do	Piece of packing ring, order 201, for use on U. S. Railroad Administration engines.
41	A	do	Do.
42–43, inclusive	D	17 504	Two entire packing rings in service from May 22, 1918, to Dec. 15, 1918, on a Mikado or 2–8–2 superheated steam locomotive.
44–46, inclusive	C	Never in service	Three new packing rings intended for a superheated engine.
47	C	Apparently in service but no record furnished.	Small piece of packing ring which had apparently been in service.
48–49, inclusive	A	28 988	Two entire packing rings which made "fair" service on engine 5107, Pacific type, superheated.
50–55, inclusive	E	1516	Pieces of packing ring giving poor service on Pacific type engine 5116, superheated.

TABLE 1—Continued

No.	Manufacturer	Service record	Description
		Miles	
56	B	11 251; 20 670; 30 793 Average 20 905	Piece of packing ring, stamped 19, from engine using saturated steam.
57	F	5349; 4324; 6085 Average 5253	Piece of packing ring, stamped No. 39, from engine using superheated steam. Scotch iron.
58	F	10 183; 10 798; [illegible] Average 9935	Piece of packing ring, stamped No. 47, from engine using superheated steam. Common iron.
59	F	22 905; 14 938 Average 18 921	Piece of packing ring, stamped No. 16, from engine using saturated steam. Common iron.
60	G	11 012; 30 427 Average 20 719	Piece of packing ring, stamped No. 25, from engine using saturated steam. Common iron.
61	G	16 918; 21 610; 11 251 Average 16 593	Piece of packing ring, stamped No. 27, from engine using saturated steam. Common iron.
62	G	7386; 6050 Average 6718	Piece of packing ring, stamped No. 55, from engine using superheated steam.
63	A	15 576; 15 943; 6832 Average 12 484	Piece of packing ring, stamped No. 33, from engine using superheated steam.
64–69, inclusive	D	Never in service	Six untested arbitration bars of packing-ring iron.
70–75, inclusive	D	do	Six chill-test blocks of packing-ring iron.
76–81, inclusive	C	do	Six untested arbitration bars of packing-ring iron.
82–87, inclusive	A	do	Do.
88–93, inclusive	A	do	Six chill-test blocks of packing-ring iron.
94–99, inclusive	B	do	Six untested arbitration bars of packing-ring iron. Rough castings.
100–103, inclusive	B	do	Four chill-test blocks of packing-ring iron.
104–109, inclusive	H	do	Six untested arbitration bars of packing-ring iron.
110–113, inclusive	H	do	Four chill-test blocks of packing-ring iron.
114–117, inclusive	I	10 000	Four pieces from packing ring from engine 2503.
118–121, inclusive	I	12 000	Four pieces from packing-ring iron, engine 2918, passenger service, using superheated steam.
122–127, inclusive	I	32 000	Six pieces of packing-ring iron, engine 2943, passenger service, using superheated steam.
128–131, inclusive	I	Never in service	Four untested arbitration test bars of packing-ring iron.

II. PREVIOUS WORK

This Bureau thought it desirable to study the previous work upon this subject, and to that end made a survey of the literature. The U. S. Railroad Administration furnished the Bureau with the results of mechanical tests and chemical analyses of a number of melts of air-furnace iron (foundry A) and also with considerable interesting information on the foundry practice of several of the makers whose products were tested in this investigation and on the service-mileage records of packing rings.

1. REVIEW OF LITERATURE

The most noteworthy contributions to this subject are in the Proceedings of the American Railway Master Mechanics' Association, now the mechanical section of the American Railroad Association. Their original specification, adopted in 1906, required the chemical composition to be within the following limits: Graphitic carbon, 2.75 to 3.25 per cent; combined carbon, 0.50 to 0.70 per cent; silicon, 1.25 to 1.60 per cent; sulphur, 0.06 to 0.10 per cent; phosphorus, 0.50 to 0.80 per cent; manganese, 0.30 to 0.60 per cent. The mechanical requirements of the same specification were a minimum tensile strength of 25 000 pounds per square inch, a minimum transverse breaking load of 3000 pounds, and a minimum deflection of 0.10 inch on the standard 1¼-inch arbitration bar of the American Society for Testing Materials.

The above specifications have undergone several revisions, until their present mechanical and chemical requirements are as indicated in Table 2. In the more recent specifications the composition of the iron is for the most part left to the foundryman, as greater dependence is placed upon the results of the transverse test. A stronger iron is required than in the earlier days.

TABLE 2.—Chemical and Mechanical Characteristics of Cast Iron for Cylinder Parts

[1918 Specifications, American Railway Master Mechanics' Association]

Chemical requirements:		
Phosphorus, maximum	per cent	0.70
Sulphur, maximum	do	.12
Transverse tests, 1¼-inch round bar, 12-inch supports:		
Strength (castings ⅝ inch or less thick), minimum	pounds	3200
Strength (castings over ⅝ inch thick), minimum	do	3500
Deflection (both cases), minimum	inch	.09
Chill in 1¾ by 2 inch block:		
For castings ⅝ inch or less thick, minimum	do	1/16
For castings over ⅝ inch thick	do	⅛

In 1916 a committee of the American Railway Master Mechanics' Association [1] appointed to investigate cast iron for cylinder parts reported in part as follows:

That gun iron (air-furnace iron) is generally considered to be efficient, economical, and for these reasons desirable, can not be doubted, considering the fact that 80 per cent of the roads reporting are using this product in their superheater locomotives for some of the parts mentioned; therefore, the committee does not hesitate to recommend its use for piston-valve bushings, piston-valve packing rings, and pistons or piston bull rings.

[1] Proc. Am. Railway Master Mechanics Assoc., 49, p. 234; 1916.

Moldenke [2] offers the following explanation of the origin of the term "gun iron":

> This is essentially an air-furnace iron, and, having been put into cast-iron guns from the earliest times, has retained that appellation. At present this grade of metal is used for the most important classes of work, and, being expensive to produce, only the best of pig irons enter into it as a rule.

With regard to cast iron for locomotive cylinders, Moldenke [3] remarks:

> For the latest type of compound engines operated with superheated steam at very high temperatures only the very best of irons should be used, and preferably melted in the air furnace. The metal must be strong and sound, as otherwise leaks develop, which cause loss of power to a serious extent. Moreover, the iron is under very high temperatures and, unless low in graphite, will give serious trouble from "growing."

Hurst [4] has published an interesting paper, which, however, deals mostly with cast iron for cylinders and pistons for internal combustion engines, where the temperatures are considerably higher than in superheated steam locomotive cylinders.

2. DATA FURNISHED BY U. S. RAILROAD ADMINISTRATION

(*a*) FOUNDRY PRACTICE.—The U. S. Railroad Administration advised this Bureau that of the nine foundries supplying material for this investigation, A is the only maker which used the air furnace; all others use the cupola process, as far as could be ascertained.

The cupola is a vertical-shaft furnace lined with refractory material; an opening is provided in the side of the furnace near the top for charging fuel and iron. An air blast is introduced near the bottom, where the molten metal is tapped. In this process of manufacture the iron is in intimate contact with the fuel and absorbs from it such objectionable impurities as sulphur. The air furnace, on the other hand, is of the reverberatory type, and the fire is at one end and the chimney at the other end of the hearth. The flame sweeps across the furnace and furnishes the heat necessary to melt the metal. It is obvious that the iron and fuel are not in as close contact as in the cupola. Other methods of melting in use by the gray-iron foundry are the crucible, the open-hearth, and the electric furnace. As no very considerable percentage of gray-iron castings are made by any of these processes, they are not described in this paper. The electric furnace, however, is rapidly coming to the foreground in the iron foundry.

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[2] Richard Moldenke, Principles of Iron Founding, p. 191; 1917. McGraw Hill Book Co.

[3] Principles of Iron Founding, p. 193; 1917.

[4] J. E. Hurst, Growth of Cast Iron by Repeated Heating, Proc. Staffordshire Iron and Steel Inst., **33**, pp. 26–54; 1918. Also, Foundry, **46**, pp. 227–229; 1918.

Manufacturer A, who, as previously stated, uses the air furnace, claims that no steel or ferroalloys are added for the purpose of furnishing strength and that no difficulty is experienced in obtaining a transverse strength of arbitration bar of 4000 pounds, or a tensile strength of 32 000 pounds per square inch; also that the sulphur content seldom exceeds 0.06 per cent. Foundry C states that his practice is to add 2 per cent of an 80 per cent ferrosilicon to approximately 4000 pounds of metal, also claiming that great care should be exercised in skimming and gating when pouring packing rings and bushings. Manufacturer D, who states that he uses 15 per cent of steel scrap in the cupola, furnishes the following additional information:

Cylinder and valve rings are made in the form of pot castings in green-sand molds. Piston-valve bushings are also cast in green-sand molds, and the steam ports are cored with the admission and the exhaust ports both on one core, and the mold is poured at the center. The cylinder bushings are cast on end with dry-sand molds, and the pouring of metal is done as rapidly as possible; the bushings are cast without steam ports.

(*b*) Tests Made by U. S. Railroad Administration.—One of the railroad inspectors tested samples from 23 casts of air-furnace iron made between July 15, 1918, and March 10, 1919, with the results shown in Table 3.

TABLE 3.—Results of Air-Furnace Iron Tests by U. S. Railroad Administration

Items determined	Average	Maximum	Minimum
Transverse strength of 1¼-inch arbitration bar in pounds, using 12-inch supports	4300	4939	3583
Transverse modulus of rupture of arbitration bar, pounds per square inch	67 300	77 200	56 000
Deflection, inch	.117	.144	.089
Tensile strength, pounds per square inch	33 400	38 000	27 000
Total carbon, per cent	3.23	3.50	2.84
Graphitic carbon, per cent	2.38	2.73	1.94
Combined carbon, per cent	.84	1.02	.59
Silicon, per cent	1.26	1.57	.95
Sulphur, per cent	.052	.100	.036
Phosphorus, per cent	.39	.49	.30
Manganese, per cent	.40	.52	.26

In these heats the strength of arbitration bar was below 4000 pounds in only two instances (3583 and 3720 pounds), and the sulphur content only twice exceeded 0.06 per cent (0.062 and 0.100 per cent).

Several of the railroads made service tests of packing rings, and these records, together with the samples, were kindly furnished

to the Bureau through the U. S. Railroad Administration. At this point it would, perhaps, be desirable to emphasize the fact that many other factors besides the quality of the iron have considerable bearing upon the results of these tests; such factors are design, lubrication, method of handling locomotive, level or hilly country, good and bad water districts, etc. As a consequence, these facts are to be borne in mind in endeavoring to establish a correlation between laboratory and service tests.

III. INVESTIGATION AT BUREAU OF STANDARDS

1. MECHANICAL TESTS

The mechanical tests made at the Bureau of Standards consisted of transverse, tension, hardness, and fracture of chill-test specimens furnished by the U. S. Railroad Administration.

(*a*) TRANSVERSE TESTS.—*Arbitration Bars.*—Thirty-four arbitration bars, ordered made in accordance with the American Society for Testing Materials standards, from six different manufacturers, were tested (Tables 4 and 5). The foundries were A, H, B, C, I, and D, the preceding order indicating the quality, the first-named being the highest in transverse strength in terms of the modulus of rupture in pounds per square inch. The bars from H showed a higher transverse breaking load in pounds than those from A, but, when the variation in diameter of bar is taken into consideration, the latter is found to be the stronger material. The bars from foundries C and D were unfortunately machined before testing, and, as a consequence, the average strength of the bar is decreased because of the absence of skin hardness. The iron from both sources, however, was of low strength, which fact was amply demonstrated by subsequent tests. The iron from foundry I was irregular in quality, while that from D and H was spongy.

TABLE 4.—Transverse Tests of Unmachined Arbitration Bars

[Span 12-inch, centrally applied load]

No.	Manufacturer	Diameter	Breaking load	Modulus of rupture	Maximum deflection
		Inches	Pounds	Lbs./in.²	Inch
82	A	1.23	3836	63 900	0.117
83	A	1.22	3900	65 600	.110
84	A	1.23	4080	67 000	.122
85	A	1.22	4220	71 000	.136
86	A	1.22	4414	74 200	.132
87	A	1.23	4116	68 600	.132
Average		1.23	4094	68 100	.125

TABLE 4—Continued

No.	Manufacturer	Diameter	Breaking load	Modulus of rupture	Maximum deflection
		Inches	Pounds	Lbs./in.²	Inch
94	B	1.25	3600	56 300	0.101
95	B	1.27	4280	63 900	.138
96	B	1.25	4210	65 800	.137
97	B	1.25	3986	62 300	.119
98	B	1.27	4150	62 000	.124
99	B	1.25	3480	54 400	.095
Average		1.26	3953	60 800	.119
104	H	1.28	4320	63 000	.099
105	H	1.29	4320	61 500	.104
106	H	1.27	4260	63 600	.111
107	H	1.26	4250	64 900	.118
108	H	1.27	4380	65 100	.115
109	H	1.31	4430	60 200	.102
Average		1.28	4323	63 100	.108
128	I	1.25	4140	64 800	.125
129	I	1.27	2500	37 300	.075
130	I	1.26	3600	55 000	.104
131	I	1.27	3160	47 100	.097
Average		1.26	3325	51 100	.100

TABLE 5.—Transverse Tests of Machined Arbitration Bars

[Span 12-inch, centrally applied load]

No.	Manufacturer	Cross section		Breaking load	Modulus of rupture
		Width	Height		
		Inch	Inch	Pounds	Lbs./in.²
64	D	0.865	0.860	1924	45 100
65	D	.875	.873	1714	46 200
66	D	.856	.865	1588	44 400
67	D	.857	.858	1592	44 700
68	D	.864	.860	1794	50 800
69	D	.854	.859	1630	46 600
Average		.862	.862	1652	46 300
76	C	.854	.851	1950	53 800
77	C	.852	.853	1565	45 500
78	C	.854	.854	1864	53 800
79	C	.853	.852	1820	52 900
80	C	.852	.852	1830	53 200
81	C	.863	.850	1650	47 600
Average		.855	.852	1753	51 100

Packing Rings.—When the packing rings were sufficiently large in size, specimens for small transverse tests were machined from them, as indicated in Fig. 1. The reader is referred to Table 6 for the results of the tests, together with the service records.

It is not to be expected that these results will be similar to those on unmachined arbitration bars, because of the absence of skin or surface hardness. The rings from manufacturer A, which gave fairly good service, had a transverse strength of 48 900 pounds per square inch; B, 47 400 pounds per square inch; C, 46 800

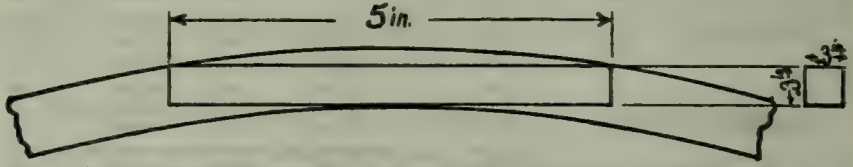

FIG. 1.—*Method of cutting small transverse test pieces from packing ring*

pounds per square inch; and D, 40 600 pounds per square inch. It is apparent from these tests that the transverse strength of the ring from foundry B, which gave the excellent service record of 93 000 miles, was not exceptional.

TABLE 6.—Transverse Tests of Packing Rings

[Span 4 inches, centrally applied load]

No.	Manufacturer	Cross section		Modulus of rupture	Maximum deflection	Service record
		Width	Height			
		Inch	Inch	Lbs./in.²	Inch	Miles
42	D	0.311	0.309	41 200	0.070	17 504
43	D	.308	.309	40 000	.073	17 504
Average				40 600		
44	C	.304	.307	45 300	.085	
44	C	.313	.307	44 400	.073	
45	C	.304	.305	53 500	.072	
45	C	.306	.306	44 600	.071	
46	C	.313	.312	45 300	.074	
46	C	.314	.313	42 900	.071	
47	C	.313	.308	51 800	.068	
Average				46 800		
48	A	.297	.312	50 000	.092	28 988
48	A	.312	.306	50 300	.074	28 988
49	A	.313	.308	46 500	.077	28 988
Average				48 900		
2	B	.298	.302	46 200	.071	93 000
24	B	.287	.292	47 100	.073	93 000
56	B	.276	.312	48 900	.070	20 905
Average				47 400		

(*b*) TENSION TESTS.—*Arbitration Bars.*—After breaking the arbitration bars in the transverse tests the broken pieces were used to determine the tensile strength of the material. The results of the tests of the products of six of the foundries are summarized

in Table 7. The iron from A, as in practically all of the tests, showed some slight superiority, with an average tensile strength of 34 600 pounds per square inch. The others in order of decreasing strength were B, 33 500 pounds per square inch; I, 32 050 pounds per square inch; H, 30 700 pounds per square inch; D, 25 750 pounds per square inch; and C, 25 050 pounds per square inch.

TABLE 7.—Tension Tests of Arbitration Bars

No.	Manufacturer	Diameter	Tensile strength	No.	Manufacturer	Diameter	Tensile strength
		Inch	Lbs./in.²			Inch	Lbs./in.²
82	A	0.505	33 300	94	B	0.505	32 500
84	A	.503	35 900	97	B	.502	34 100
Average		.505	34 600	Average		.503	33 300
64	D	.501	24 200	106	H	.503	30 600
69	D	.505	27 200	108	H	.503	30 800
Average		.503	25 750	Average		.503	30 700
77	C	.505	26 250	128	I	.503	34 600
79	C	.505	23 900	129	I	.505	30 600
Average		.505	25 050	130	I	.505	32 100
				131	I	.503	30 900
				Average		.504	32 050

Packing Rings.—Tension tests were made from packing rings furnished by foundries A, B, F, and G, and the results, together with service records, are to be found in Table 8. The ring from foundry B, which gave excellent service, also had the highest tensile strength; namely, 25 450 pounds per square inch. The other rings, all of which gave poor results in the service tests, were likewise inferior in tensile strength. From these tests, at least, there appears to be some more or less definite relation between laboratory and service tests.

TABLE 8.—Tension Tests of Packing Rings

No.	Manufacturer	Diameter	Tensile strength	Service record
		Inch	Lbs./in.²	Miles
5	B	0.293	27 500	93 000
7	B	.300	23 400	93 000
13	B	.299	25 500	93 000
Average			25 450	
57	F	.505	18 300	5253
58	F	.505	18 500	9935
Average			18 500	
62	G	.505	21 900	6718
63	A	.501	16 700	12 484

(*c*) BRINELL HARDNESS TESTS.—*Arbitration bars.*—Brinell hardness tests, using a 10 mm ball with a pressure of 3000 kg, sustained for 30 seconds, were made on flat, longitudinal sections of the arbitration bars. Approximately one-fourth of an inch of material was removed by the shaper to obtain a surface for the Brinell tests. In Table 9 it will be seen that the iron from A was hardest, with a Brinell hardness numeral of 213, H and I next, each with a hardness of 211, and that C was softest, with a Brinell hardness numeral of 203. As there is a variation of only 10 points from the maximum to minimum hardness, it is concluded that, in view of the widely varying quality of the irons as indicated by other tests, the Brinell test does not satisfactorily measure the quality of iron for cylinder parts.

TABLE 9.—Brinell Hardness of Arbitration Bars

[3000 kg pressure for 30 seconds, 10 mm ball]

No.	Manufacturer	Brinell hardness numerals [a]	No.	Manufacturer	Brinell hardness numerals [a]
64	D	206	94	B	207
65	D	192	95	B	211
66	D	207	96	B	207
67	D	204	97	B	206
68	D	207	98	B	226
69	D	216	99	B	197
Average		205	Average		209
76	C	206	104	H	214
77	C	192	105	H	212
78	C	206	106	H	213
79	C	206	108	H	205
80	C	205	108	H	211
81	C	204	109	H	212
Average		203	Average		211
82	A	204	128	I	210
83	A	216	129	I	207
84	A	213	130	I	219
85	A	212	131	I	210
86	A	209	Average		212
87	A	226			
Average		213			

[a] Average of 3 determinations.

TABLE 10.—Brinell Hardness of Packing Rings, Bushings, Etc.

[3000 kg pressure for 30 seconds, 10 mm ball]

No.	Manufacturer	Brinell hardness numeral	Service record	No.	Manufacturer	Brinell hardness numeral	Service record
			Miles				Miles
1	B	175	93 000	42	D	133	17 504
5	B	163	93 000	43	D	160	17 504
7	B	165	93 000	Average		147	
13	B	166	93 000				
56	B	167	20 905	51	E	146	1516
Average		167		57	F	141	5253
				58	F	159	9935
27	C	156		59	F	166	18 921
44	C	159		Average		155	
45	C	160					
46	C	163		60	G	171	20 719
47	C	167		61	G	183	16 593
Average		159		62	G	177	5718
				Average		177	
36	A	175					
37	A	153		114–117, inclusive	I	172	10 000
38	A	155		118–121, inclusive	I	158	12 000
39	A	180		122–127, inclusive	I	174	12 000
40	A	170		Average		168	
41	A	179					
48	A	151	28 988				
49	A	170	28 988				
Average		168					

Packing Rings, Bushings, and Similar Parts.—The Brinell hardness tests of packing rings, bushings, etc. (Table 10), indicate that the castings themselves are considerably softer than the arbitration bars. This is undoubtedly due to the fact that the slower rate of cooling of the former results in a higher graphitic carbon content. This is particularly true of some of the samples from foundry A, which were cylinder bushings, having a considerably larger cross-sectional area than packing rings.

(*d*) CHILL TESTS.—Chill-test specimens were received from four foundries. Foundries A and H used the standard chill-test specimen of the American Society for Testing Materials, while B and D used specimens not standard in size. Details are shown in the drawings in Fig. 2. The specimens were broken at the Bureau of Standards, four to six samples of the product of each foundry having been submitted. The iron from makers A and D showed an average depth of chill of one-half an inch, and that from B and H had a chill of approximately one-fourth of an inch.

2. CHEMICAL COMPOSITION

(*a*) ARBITRATION BARS.—Chemical analyses were made of one of each of the arbitration bars (Table 11) from each of six foundries, except in the case of I, where two bars were analyzed because of

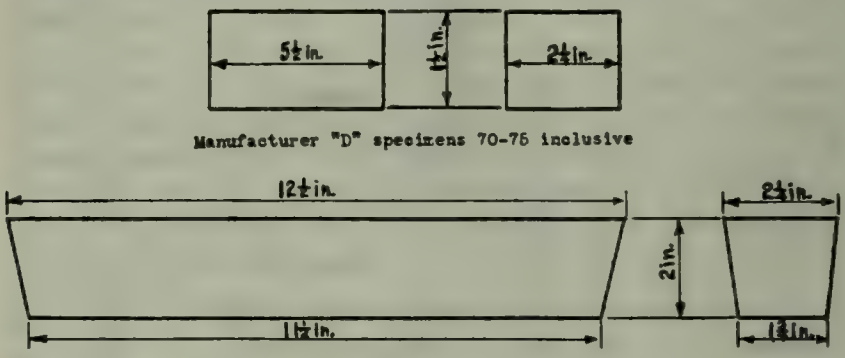

FIG. 2.—*Dimensions of three types of chill-test specimens from various manufacturers*

the widely varying mechanical properties, as shown in Table 4. The poor quality of the iron in specimen No. 129 (foundry I) is attributable to its high silicon content.

TABLE 11.—Chemical Composition of Arbitration Bars

No.	Manufacturer	Carbon			Silicon	Sulphur	Phosphorus	Manganese
		Total	Graph.	Combined				
		Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
64	D	3.36	2.55	0.81	1.42	0.093	0.48	0.61
77	C	3.36	2.40	.96	1.33	.167	.56	.39
84	A	3.30	2.39	.91	1.13	.051	.51	.36
94	B	3.30	2.50	.80	1.36	.124	.37	.90
106	H	3.32	2.42	.90	1.03	.137	.52	.54
128	I	3.26	2.37	.89	1.00	.161	.38	.63
129	I	3.31	2.66	.65	1.47	.132	.50	.53

The various bars were quite uniform in total, graphitic, and combined carbon content. The silicon and sulphur contents, however, varied within rather wide limits, the former from 1 per cent to 1.47 per cent (both samples from I) and the latter from 0.051 per cent (A) to 0.167 per cent (C). The iron from manufacturer B was the lowest in phosphorus content, with 0.37 per cent, and that from C was highest, with 0.56 per cent. Manganese varied from 0.36 per cent in the product from A to 0.90 per cent in the material from foundry B.

It is generally recognized that there should be at least three times the percentage of manganese as sulphur present; otherwise, sulphur will combine with iron instead of manganese, and brittle material will result. It will be observed that in the iron from C there is approximately only twice as much manganese as sulphur present. This condition, together with the high combined carbon content, accounts, in all probability, for the poor showing of this iron in the mechanical tests (Tables 5 and 7).

(*b*) Packing Rings, Bushings, and Similar Parts.—In general (Table 12), the silicon, and naturally the graphitic carbon, is higher in the packing rings, bushings, and similar castings than in the arbitration bars. The ring from B, which gave 93 000 miles in service, showed no unusual characteristics in chemical composition. The rings from foundry C were high in phosphorus and sulphur, as in the case of the arbitration bars. The castings from B were low in sulphur, and the ring which had a service record of 28 988 miles was rather low in combined carbon content. D's rings were high in sulphur and graphitic carbon. The latter condition (high graphitic carbon), of course makes too soft a ring for prolonged wear. The locomotive packing ring from foundry E, which gave a service of only 1516 miles, was very high in silicon (2.84 per cent), and consequently low in combined carbon (0.33 per cent). The rings from I (32 000 miles of service) were fairly low in graphitic carbon content.

TABLE 12.—Chemical Composition of Packing Rings, Bushings, Etc.

No.	Manufacturer	Carbon			Silicon	Sulphur	Phosphorus	Manganese	Service record
		Total	Graph.	Combined					
		Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Miles
1	B	3.46	2.79	0.67	1.68	0.120	0.260	0.56	93 000
5	B	3.39	2.80	.59	1.67	.140	.260	.57	93 000
7	B	3.53	2.91	.62	1.67	.150	.250	.57	93 000
13	B	3.43	2.69	.74	1.69	.130	.250	.58	93 000
56	B	3.08	2.67	.41	1.70	.124	.825	.43	20 905
27	C	3.36	2.42	.94	1.47	.190	.310	.42	.....
44	C	3.57	2.88	.69	1.51	.130	.601	.51	.....
45	C	3.45	2.84	.61	1.95	.130	.582	.51	.....
47	C	3.44	2.87	.57	1.46	.136	.639	.56	.....
29	A	3.33	2.42	.91	1.21	.060	.250	.38	.....
36	A	3.24	2.40	.84	1.06	.071	.220	.24	.....
37	A	3.37	2.75	.62	1.24	.068	.260	.28	.....
38	A	3.31	2.90	.41	1.50	.068	.230	.54	.....
39	A	3.24	2.65	.59	1.21	.150	.220	.45	.....
40	A	3.31	2.56	.75	.85	.055	.220	.40	.....
41	A	3.28	2.55	.73	.87	.062	.210	.61	.....
48	A	3.46	2.91	.55	1.52	.062	.502	.45	28 988
63	A	3.51	2.84	.67	1.40	.102	.440	.38	12 484
42	D	3.47	3.12	.35	1.83	.151	.558	.56	17 504
43	D	3.50	3.10	.40	1.94	.165	.704	.55	17 504
50	E	3.34	3.01	.33	2.84	.068	.675	.55	1516
57	F	3.40	2.74	.66	1.58	.117	.410	.35	5253
58	F	3.24	2.73	.51	2.14	.119	.855	.32	9935
62	G	3.47	2.68	.79	1.33	.106	.380	.42	6718
116	I	3.22	2.53	.69	1.83	.112	.670	.34	10 000
117	I	3.11	2.54	.57	1.78	.116	.670	.34	10 000
118	I	3.16	2.54	.62	1.22	.140	.360	.52	11 000
121	I	3.29	2.53	.76	1.25	.140	.370	.52	12 000
122	I	3.35	2.57	.78	1.96	.087	.740	.44	31 000
125	I	3.38	2.76	.62	1.95	.091	.760	.43	32 000

3. METALLOGRAPHIC EXAMINATION

Specimens from packing rings made by B (No. 5, 93 000 miles), A (No. 48, 28 988 miles; No. 63, 12 484 miles), D (No. 42, 17 504 miles), G (No. 62, 6718 miles), and E (No. 50, 1516 miles) were examined microscopically.

The samples were polished and examined microscopically both before and after etching. Heat tinting was found to be very suitable for revealing the irregularly shaped masses of phosphide eutectic and at the same time allowing the black flakes of graphite to stand out clearly. Micrographs were taken only of typical

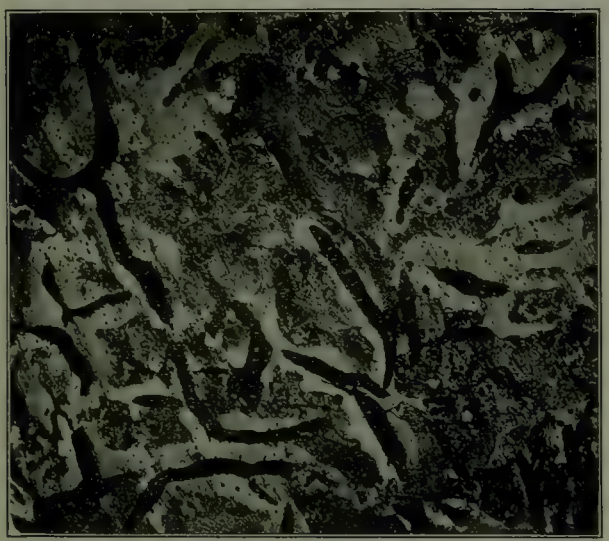

FIG. 3.—*Specimen No. 5*

Manufacturer "B" (93 000 miles service). Etched with 2 per cent nitric acid. ×100

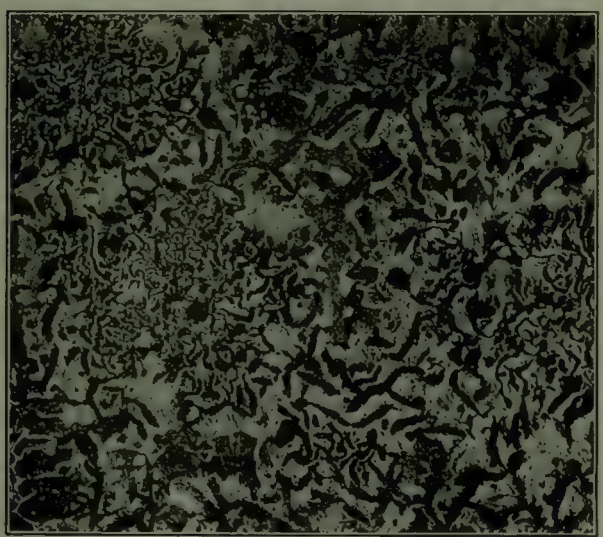

FIG. 4.—*Specimen No. 50*

Manufacturer "E" (1516 miles service). Etched with 2 per cent nitric acid. ×100

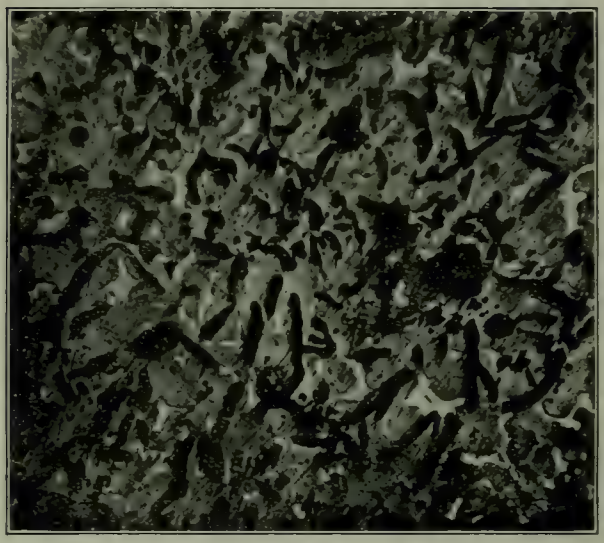

FIG. 5.—*Specimen No. 42*

Manufacturer "D" (17 504 miles service). Heat tinted. ×50

FIG. 6.—*Specimen No. 42*

Manufacturer "D" (17 504 miles service). Etched with 2 per cent nitric acid. ×100

samples selected for purposes of illustration. Fifty diameters magnification was used to show the size, distribution, and amount of graphite flakes, and 100 diameters to show the constitution of the matrix.

Some attempt was made to correlate the microstructure with the endurance in service, but, due to the many variable factors, it was realized that any very close comparisons were apt to be misleading. The inhomogeneity of cast iron made it somewhat doubtful that the small sample used for metallographic examination was representative of the whole. In some instances, however, with the aid of the principles governing the constitution of gray cast iron, the samples could be approximately classified, and the service records bore out fairly well the predictions that could be made of mechanical qualities from a study of the microstructure.

In Figs. 3 and 4 are shown samples Nos. 5 and 50 after etching with 2 per cent nitric acid. Fig. 3 is the microstructure of the ring which gave 93 000 miles of service, while Fig. 4 shows the ring which gave only 1516 miles' service. The difference between them is striking. In Fig. 3 the gray areas are pearlite, the pure white is ferrite, the irregular speckled masses are phosphide, and the large, black flakes are graphite. In Fig. 4 the white constituent, rather large in amount, is ferrite, the gray areas forming the easily visible network are pearlite, the irregular lighter-colored masses are phosphide, and the small, black flakes are graphite. As might be inferred from the chemical composition (No. 5, 0.59 per cent combined carbon; No. 50, 0.33 per cent combined carbon) the matrix of the former is largely pearlitic, with very little ferrite, while the reverse is true in the latter. The rate of cooling in the latter sample (No. 50) was the more rapid, and thus the finely divided condition of the graphite is to be explained. The very high silicon content of 2.48 per cent accounts for the high graphitic carbon content. This resulted in a soft iron, which, together with the physical unsoundness of the material as revealed by microscopical examination, accounted for the poor service record of 1516 miles. B's ring, on the other hand, while higher in combined carbon (pearlite) and lower in graphite or free carbon, under the microscope shows no explanation for the extraordinarily long service obtained.

The microstructure of the ring from D (No. 42, 17 504 miles) is shown in Figs. 5 and 6. While the combined carbon content is low the distribution of the various constituents is much more uniform than in Fig. 4. The ring, however, was too soft to give

good wearing qualities. In Fig. 7 is shown the structure of A's ring, sample No. 48, which gave 28 988 miles of service. It does not materially differ in microstructure from the other rings giving fair service; it is, in fact, as indicated by Brinell hardness test and chemical composition, somewhat too low in combined carbon for maximum wear. Fig. 8 shows the microstructure of a ring made by G (6718 miles). The distribution of the graphite is quite uniform, and from a metallographic point of view it is impossible to account for the poor results reported in service.

The microscope is most useful in determining the size, amount, and distribution of graphite flakes, amount of combined carbon, and to some extent the physical soundness of the castings.

IV. COMPARISON OF PROPERTIES OF AIR-FURNACE AND CUPOLA CAST IRON

From a study of the data given in the various tables of tests, it will be observed that the air-furnace iron is somewhat more uniform in character and of generally better quality than cupola iron. A comparison of the two kinds of iron, bearing out this conclusion, and which was computed from all tests of arbitration bars mentioned in this paper, is given in Table 13.

TABLE 13.—Properties of Air-Furnace Iron and of Cupola Cast Iron

Process of manufacture	Transverse test, modulus of rupture			Tensile strength		
	Average	Maximum	Minimum	Average	Maximum	Minimum
	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²
Air furnace	67 500	77 200	56 000	33 500	37 500	27 000
Cupola	54 700	65 800	37 300	29 700	34 600	23 900

V. CONCLUSIONS AND RECOMMENDATIONS

Conclusions are drawn and recommendations made as follows:

1. On the basis of tests made by inspectors of the U. S. Railroad Administration and this Bureau, which were substantially in agreement, it is concluded that air furnace or so-called "gun iron" is more uniform in character and on the average of somewhat better mechanical properties than cupola iron. The latter, however, often equals or even excels in mechanical properties the specimens of air-furnace iron tested in this investigation. The sulphur content of the air-furnace irons examined seldom exceeded 0.06 per cent, while the cupola irons varied in sulphur content from 0.10 to 0.17 per cent.

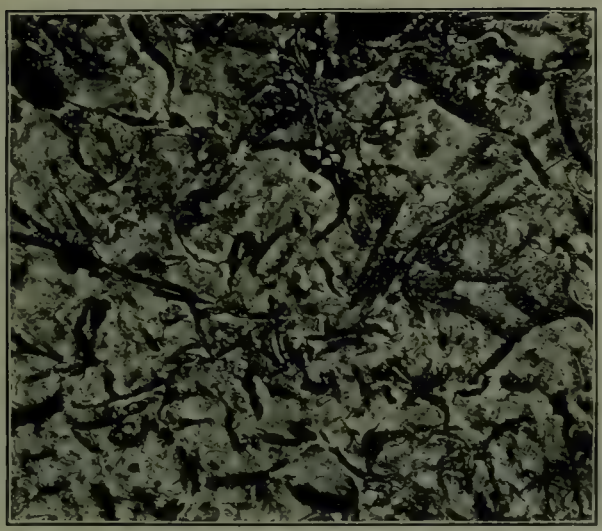

FIG. 7.—*Specimen No. 48*

Manufacturer "A" (28 988 miles service). Heat tinted. ×50

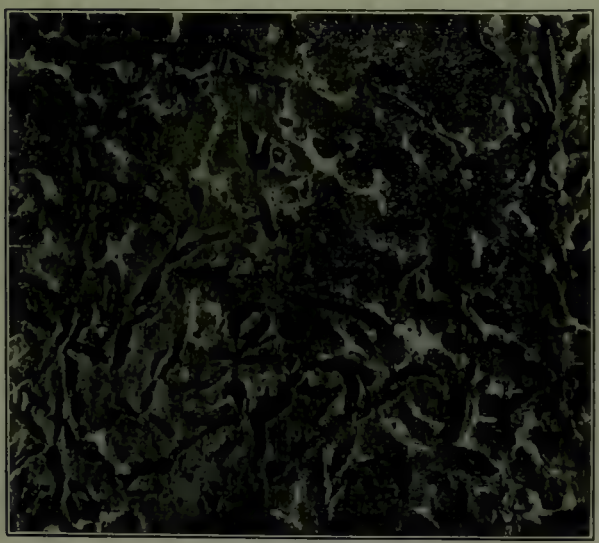

FIG. 8.—*Specimen No. 62*

Manufacturer "G" (6718 miles service). Heat tinted. ×50

2. The findings of the American Railway Master Mechanics' Association with respect to correlation of laboratory and service tests are confirmed in the present investigation. It was impossible, except in a very general way to find any correlation between the quality of the iron as developed by laboratory tests and the mileage obtained in service. This is explained by the fact that many other factors besides the quality of the iron enter into consideration in the service results; namely, design, lubrication, method of handling the locomotive, topography of the country, character of water used in locomotive, etc. The ring from foundry B, which gave the exceptionally good service of 93 000 miles, showed no unusual properties in laboratory tests. It is for the reasons cited above that the conclusions and recommendations of this paper are based essentially upon the results of laboratory tests.

3. The present specifications of the American Railway Master Mechanics' Association are somewhat lax in the requirements for mechanical properties. It is recommended that the transverse-strength requirements of 1¼-inch arbitration bar be increased from 3200 to 3500 pounds for castings one-half of an inch or less in thickness, and from 3500 to 3800 pounds for castings over one-half of an inch in thickness. It is further recommended that the minimum deflection requirements for both cases be increased from 0.09 to 0.11 inch. The division line of the casting thickness is changed from five-eighths of an inch of the American Railway Master Mechanics' Association, to one-half of an inch in order to conform to the recognized standards of the American Society for Testing Materials.

4. It is preferable to leave the chemical composition and the melting process used to the manufacturer, depending for the most part on the mechanical tests, and of these primarily upon the transverse test. The existing specifications allow a maximum of 0.70 per cent phosphorus and 0.12 per cent sulphur; there are no developments in this investigation which would warrant a revision of the maximum permissible amounts of these elements.

Acknowledgment is due to Capt. S. N. Petrenko and T. W. Greene for the mechanical tests and also to S. Epstein for the metallographic work. The cooperation of the chemistry division of the Bureau of Standards in making the chemical analyses is likewise acknowledged.

PART 2

PROPOSED SPECIFICATION[5] FOR CAST-IRON CYLINDER PARTS FOR LOCOMOTIVES

I. SCOPE

These specifications cover cast iron for piston-valve bushings, piston-valve packing rings, piston-valve bull rings, cylinder bushings, piston packing rings, and piston-head or bull rings.

II. MANUFACTURE

1. PROCESS

The castings shall be made from good quality close-grained iron.

III. CHEMICAL PROPERTIES AND TESTS

1. CHEMICAL COMPOSITION

Drillings taken from the fractured ends of the transverse-test bars shall conform to the following limits in chemical composition:

	Per cent
Phosphorus, not over	0.70
Sulphur, not over	.12

IV. PHYSICAL PROPERTIES AND TESTS

1. TRANSVERSE TEST

When placed horizontally upon supports 12 inches apart and tested under a centrally applied load, the arbitration-test bars specified in Section IV–3*a* shall show an average transverse strength of not less than 3500 pounds for castings one-half inch or less in thickness, and an average transverse strength of not less than 3800 pounds for castings over one-half inch in thickness; and the deflection for either thickness of material shall not be less than 0.11 inch. The rate of application of the load shall be from 20 to 40 seconds for a deflection of 0.10 inch.

2. CHILL TEST

Before pouring, a sample of the iron shall be taken and chilled in a cast-iron mold, as specified in Section IV–3*b*. The sample shall be allowed to cool in the mold until it is dark red or almost

[5] Prepared at the request of the U. S. Railroad Administration to embody the results of this and other investigations.

black, when it may be knocked out and quenched in water. The sample, on being broken, must show a close-grained gray iron, with a well-defined border of white iron at the bottom of the fracture. The depth of the white iron must not be less than one-sixteenth of an inch as measured at the center line, for castings one-half inch or less in thickness, nor less than one-eighth of an inch for castings over one-half inch in thickness.

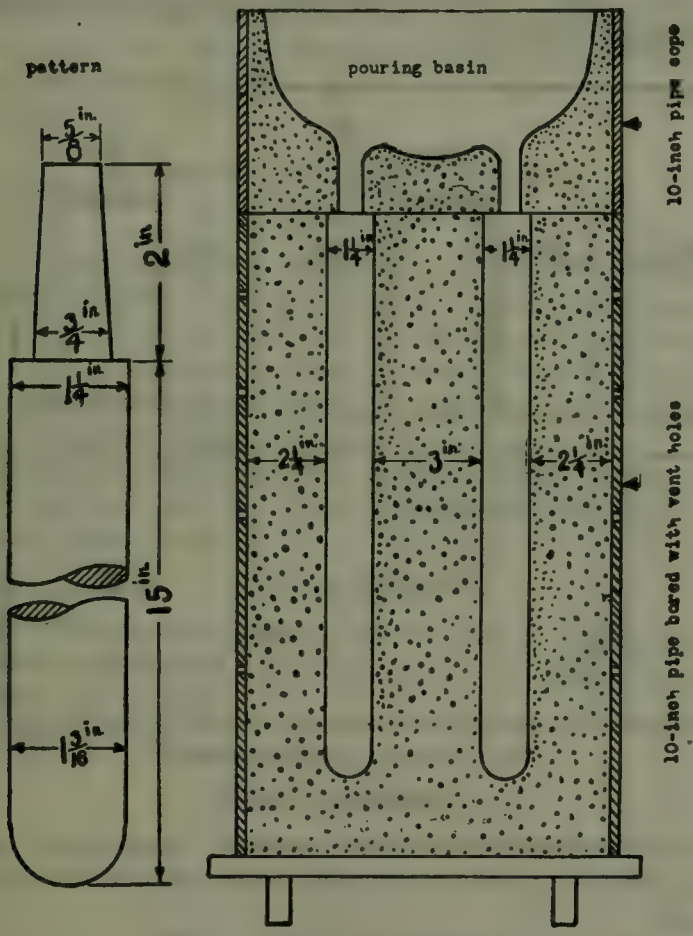

FIG. 9.—*Mold for arbitration test bar*

3. MOLDS FOR TEST SPECIMENS

(*a*) ARBITRATION BAR.—The mold for the bars is as shown in Fig. 9. The bottom of the bar is one-sixteenth of an inch smaller in diameter than the top to allow for draft and strain of pouring. The diameter of the test bar shall not vary more than 0.02 inch above or below the specified size. The flask is to be rammed up

with green molding sand, a little damper than usual, well mixed and put through a No. 8 sieve, with a mixture of 1 to 12 bituminous facing. The mold shall be rammed evenly and fairly hard, thoroughly dried, and not cast until it is cold. The test bar shall not be removed from the mold until it is cold enough to be handled. It shall not be rumbled or otherwise treated, being simply brushed off before testing.

(*b*) CHILL TEST.—The form and dimensions of the mold shall be in accordance with Fig. 10.

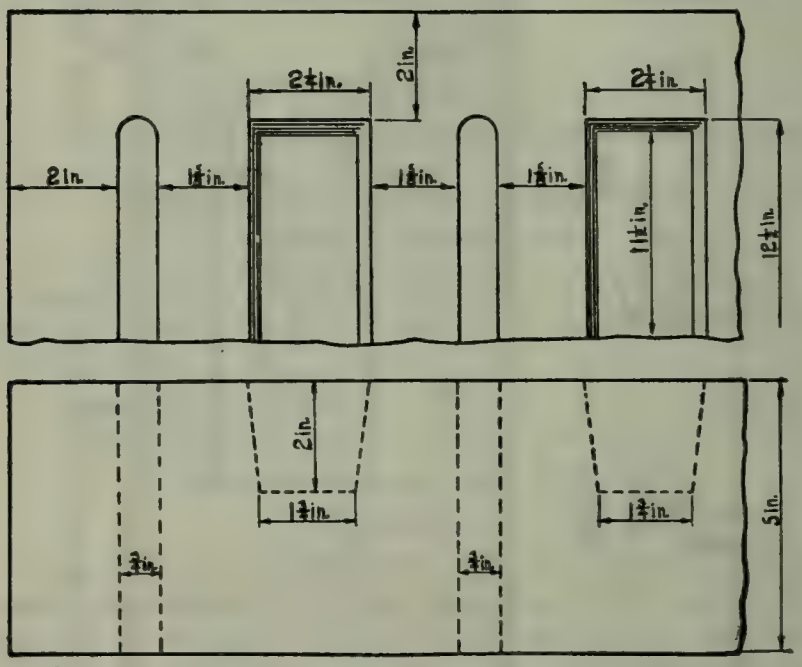

FIG. 10.—*Mold for chill-test specimen*

4. NUMBER OF TESTS

(*a*) Two arbitration-test bars, cast as specified in section IV–3*a*, shall be poured from each ladle used for one or more castings.

(*b*) One chill test, cast as specified in Section IV–3*b*, shall be poured from each ladle of metal used for one or more castings. The chill-test specimens may be cast in adjacent molds, but in such cases a space must be provided between the molds. (See Fig. 10.)

V. WORKMANSHIP AND FINISH

1. CHARACTER OF CASTINGS

Castings shall be smooth, well cleaned, free from shrinkage cracks and from other defects sufficiently extensive to impair their value, and must finish to blue-print size.

2. MARKING

Each casting when so specified shall have cast on it, in raised letters and figures, marks designating the maker, the date of casting, the serial and pattern numbers, and other marks specified by the purchaser.

VI. INSPECTION AND REJECTION

1. INSPECTION

(*a*) The purchaser, or his inspector, shall be given a reasonable opportunity to witness the pouring of the castings and test specimens, as well as to be present when the mechanical tests are made.

(*b*) In case the inspector is not present to witness the pouring of the castings and test specimens, the manufacturer will hold the latter for test by the inspector upon his arrival.

(*c*) Inspection shall be made at the place of manufacture, and the manufacturer shall also furnish the facilities for making the mechanical tests, if desired.

2. REJECTION

Unless otherwise specified, any rejection based on tests made in accordance with Section IV–1 shall be reported within five days from receipt of samples.

WASHINGTON, April 30, 1920.

DEPARTMENT OF COMMERCE

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TESTS OF BOND RESISTANCE BETWEEN CONCRETE AND STEEL

BY

W. A. SLATER, Engineer Physicist
Bureau of Standards

F. E. RICHART, Structural Engineer
G. G. SCOFIELD, Structural Engineer
Emergency Fleet Corporation

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By W. A. Slater, F. E. Richart, and G. G. Scofield

CONTENTS

	Page
I. Introduction	6
1. Purpose and scope	6
2. Acknowledgment	8
II. Pull-out tests with bars treated to prevent corrosion	9
1. Preliminary	9
2. Description of specimen	9
3. Anticorrosive coatings	10
4. Materials	11
5. Making of specimens	13
6. Auxiliary specimens	13
7. Storage of specimens	14
8. Method of testing	15
9. Nature of the bond resistance of a coated bar	16
10. General characteristics of results	17
11. Action of individual coatings	19
12. Comparison of plain and deformed bars	30
13. Effect of variations in conditions of storage	30
14. Effect of manner of applying coatings	31
15. Summary	32
III. Tests of reinforced concrete beams with tension bars lapped in a region of uniform bending moment	33
1. Preliminary	33
2. Description of specimens	33
3. Materials	35
4. Making of specimens	35
5. Method of testing	36
6. Phenomena of the tests	37
7. General results	47
8. Distribution of tensile stress among bars	47
9. Distribution of tensile stress along bars	51
10. Distribution of bond stress along bars	53
11. Slip of bars	54
12. Length of lap required for beams with through bars	56
13. Effect of variation of area of through bars on length of lap required	59
14. Summary of results	59
IV. Tests on stirrup pull-out specimens	60
1. Preliminary	60
2. Description of specimens	61
3. Making of specimens	62
4. Auxiliary specimens	63
5. Method of testing	63
6. Results of tests	64

LIST OF TABLES

		Page
1.	System of reference symbols used to indicate the nature of the coatings, methods of application, and conditions of storage	10
2.	Classification of pull-out test specimens	11
3.	Mechanical analysis of sand	12
4.	Physical properties of steel	12
5.	Composition of artificial sea water	14
6.	Bond stresses at specified slips in percentage of maximum bond stresses	18
7.	Bond stresses for coated bars in percentage of bond stresses for uncoated bars	19
8.	Characteristic action of coatings in bond tests and corrosion tests	28
9.	Comparison of bond stresses in plain and deformed bars	30
10.	Effect of conditions of storage on bond strength	31
11.	Relative values of bond resistance at 0.01-inch slip—effect of various methods of applying coating	32
12.	Reinforcement of beams with lapped bars	34
13.	Physical properties of reinforcing steel used in beam tests	35
14.	Strength of beams and control cylinders	36
15.	Tests of stirrup pull-out specimens	65

LIST OF FIGURES

1.	Deformed bar used in pull-out tests	facing	12
2.	Pull-out specimen in machine in position for testing		15
3.	Bond stresses developed with different coatings under various conditions of storage		20

BOND-SLIP DIAGRAMS FOR PLAIN AND DEFORMED BARS UNDER VARIOUS CONDITIONS OF STORAGE

4.	Uncoated bars	21
5.	Coating B, coal tar	21
6.	Coating C, asphaltic	21
7.	Coating D, asphaltic	21
8.	Coating E, ferric oxide	21
9.	Coating F, coal tar	21
10.	Coating G, asphaltic	22
11.	Coating H, asphaltic	22
12.	Coating I, ferric oxide	22
13.	Coating J, red lead	22
14.	Coating K, ferric oxide	22
15.	Coating L, ferric oxide	22
16.	Coating M, asphaltic	23
17.	Coating N, coal tar	23
18.	Coating O, galvanized	23
19.	Coating P, sherardized	23
20.	Coating Q, metal spray	24
21.	Coating V, phosphate film	24
22.	Coating X, electro galvanized	24

BOND-SLIP DIAGRAMS FOR PLAIN AND DEFORMED BARS UNDER VARIOUS METHODS OF APPLYING COATINGS

23.	Coating C, stored in damp sand 28 days	25
24.	Coating C, stored in damp sand one month, salt water five months	25
25.	Coating C, stored in damp sand one month, salt water and air five months	25
26.	Coating C, stored in damp sand one month, air five months	25

Page

27. Coating H, stored in damp sand 28 days 26
28. Coating H, stored in damp sand one month, salt water five months 26
29. Coating H, stored in damp sand one month, salt water and air five months .. 26
30. Coating H, stored in damp sand one month, air five months 26
31. Coating K, stored in damp sand 28 days 26
32. Coating K, stored in damp sand one month, salt water five months 26
33. Coating K, stored in damp sand one month, salt water and air five months ... 27
34. Coating K, stored in damp sand one month, air five months 27
35. Coating N, stored in damp sand 28 days 27
36. Coating N, stored in damp sand one month, salt water five months 27
37. Coating N, stored in damp sand one month, salt water and air five months ... 27
38. Coating N, stored in damp sand one month, air five months 27
39. Details of test beams with lapped bars 34
40. Beam in testing machine ready for application of load ..... facing 37

LOAD-STRESS DIAGRAMS

41. Beam 8AAA1; lapped bars only; sections A–F 39
42. Beam 8AAA1; lapped bars only; sections G–K 39
43. Beam 8AAB1; one-half inch through and one-half inch lapped bars; sections A–C 40
44. Beam 8AAB1; one-half inch through and one-half inch lapped bars; sections D–F 40
45. Beam 8AAB1; one-half inch through and one-half inch lapped bars; sections G–H 41
46. Beam 8AAB1; one-half inch through and one-half inch lapped bars; sections I–K 41
47. Beam 8AAC1; five-eighths inch through and one-half inch lapped bars; sections A–C 43
48. Beam 8AAC1; five-eighths inch through and one-half inch lapped bars; sections D–F 43
49. Beam 8AAC1; five-eighths inch through and one-half inch lapped bars; sections G–H 44
50. Beam 8AAC1; five-eighths inch through and one-half inch lapped bars; sections I–K 44
51. Porous condition around reinforcing bars of beam 8AAD1 ..... facing 44
52. Beam 8AAD1; three-fourths inch through and one-half inch lapped bars; sections A–C 45
53. Beam 8AAD1; three-fourths inch through and one-half inch lapped bars; sections D–F 45
54. Beam 8AAD1; three-fourths inch through and one-half inch lapped bars; sections G–H 46
55. Beam 8AAD1; three-fourths inch through and one-half inch lapped bars; sections I–K 46
56. Beams after failure, showing crack development ..... facing 47

TENSILE STRESSES IN ALL BARS AT VARIOUS SECTIONS

57. Beam 8AAB1; sections A, C, and E 48
58. Beam 8AAB1; sections F, H, and K 48
59. Beam 8AAC1; sections A, C, and E 49
60. Beam 8AAC1; sections F, H, and K 49
61. Beam 8AAD1; sections A, C, and E 50
62. Beam 8AAD1; sections F, H, and K 50

Page
63. Load-deflection diagrams for all beams 51
64. Distribution of tensile stress along lapped bar in beam 8AAA1 52
65. Distribution of tensile stress along lapped bar in beams 8AAB1, 8AAC1, and 8AAD1 52
66. Distribution of bond stress along lapped bars in beams 8AAB1, 8AAC1, and 8AAD1 53
67. Distribution of bond stress along lapped bars in beams 8AAB1, 8AAC1, and 8AAD1 at higher loads 54
68. Distribution of slip along lapped bars in beams 8AAB1, 8AAC1, and 8AAD1. 55
69. Relation of tensile stress to length of lap required 57
70. Isometric view of specimen used in stirrup pull-out tests 61
71. Types of stirrups 62
72. Details of stirrup pull-out specimen 63
73. Arrangement for testing stirrup pull-out specimen 64
74. View of specimen A after failure ..... facing 66
75. View of specimen D after failure ..... facing 66
76. View of specimen B after failure ..... facing 66
77. View of specimen C after failure ..... facing 66
78. View of specimen E after failure ..... facing 66
79. View of specimen F after failure ..... facing 66
80. View of specimen G after failure ..... following 66
81. View of specimen H after failure ..... following 66
82. View of specimen I after failure ..... following 66
83. View of specimen J after failure ..... following 66
84. View of specimen K after failure ..... following 66
85. View of specimen L after failure ..... following 66

I. INTRODUCTION

1. PURPOSE AND SCOPE

This paper embodies the results of three somewhat dissimilar investigations which were made by the concrete ship section of the Emergency Fleet Corporation on the bond resistance or adhesion between concrete and reinforcing steel. One investigation was made to study the effect on bond resistance of the applications of various anticorrosive coatings on reinforcement. The second investigation was made to study the length of lap required for effective splicing of reinforcing bars in regions of high tensile stress. The third investigation was made to study the relative merits of different methods of anchoring the ends of stirrups to meet certain conditions which arise in concrete ship construction.

One of the first problems which presented itself in the design of reinforced concrete ships was to determine whether the reinforcement in the shell of a ship would be subject to corrosion, and if so, to devise methods of preventing such action. Corrosion would be dangerous because of the bursting of the concrete due to the formation of a bulky iron compound on the surface of reinforcing

bars rather than because of injury to the reinforcement itself. Experience with existing reinforced concrete structures exposed to the action of sea water or sea air has shown that a thin covering of ordinary concrete can not be expected to protect steel from corrosion indefinitely.[1] It was anticipated that this action might be prevented in a number of ways, as, for instance, (1) by using a waterproofing paint on the surface of the concrete; (2) by increasing the waterproofing qualities of the concrete itself; or (3) by using some form of protective coating on the reinforcement.

With regard to the first investigation, it was considered probable that the use of a coating on reinforcing bars would affect the bond resistance between the concrete and the bars. Hence, when tests were made to determine the effectiveness of various coatings in preventing the corrosion of steel, tests were also made to determine the effect of the same coatings on the bond resistance which could be developed. These tests were made on pull-out specimens and will be described in Section II of this paper.

The second investigation described in this paper was made because the designer needs definite knowledge as to the effectiveness of lapped bars as a tension splice. In lapping bands of reinforcement, it is important to know how many bars of a band may be safely lapped at one section, and how the length of embedment should vary with variation in the ratio of the cross-sectional area of the lapped bars to the total cross-sectional area of the steel. This is essentially a question of bond resistance, since it involves intensity and distribution of bond stresses along the length of embedment of the lapped bar.

Most of the available data on bond resistance are based on pull-out tests similar to those reported in Section II, in which the concrete block is in compression. Such tests have been shown [2] to agree reasonably well at corresponding slips with bond tests of reinforced concrete beams in which the ends of the reinforcing bars were embedded in a region either of compressive stress or of low tensile stress.

On the other hand, pull-out specimens in which the concrete block was in tension have shown [2] lower bond stresses than those specimens in which the block was in compression, and it seemed probable that bars ending in a region of high tensile stress would develop comparatively low bond stresses. To obtain evidence on

[1] R. J. Wig and L. R. Ferguson, What is the Trouble with Concrete in Sea Water, Eng. News Record, Sept. 20 to Oct. 18, 1917.

[2] D. A. Abrams, Tests of Bond Between Concrete and Steel, Bull. 71, Eng. Exp. Sta., Univ. of Illinois.

this point test beams were made in which the tension reinforcement was lapped within a region of constant bending moment. The results of these tests are presented in Section III.

In the case of the concrete tank ship, the horizontal pressure of the cargo tends to force the shell outward from the frame, thereby causing tension in the stirrups which extend from the frame into the shell. The proper anchorage of the stirrups in a thin shell is a difficult problem, and it was to furnish information on this subject that the third investigation, as described in Section IV, was carried out.

2. ACKNOWLEDGMENT

The pull-out tests discussed in Section II were made at the concrete laboratory of the Bureau of Public Roads, at Arlington, Va. The tests were made between April, 1918, and January, 1919. A. T. Goldbeck, engineer of tests of the Bureau of Public Roads, was in direct charge of the investigation. R. Harsh and W. E. Rosengarten made most of the tests. The cooperation of the Bureau of Public Roads and of these men personally was of great value in the carrying out of the research work of the concrete ship section.

The beam tests of Section III were made at the John Fritz Laboratory of the Civil Engineering Department, Lehigh University, in December, 1918. Acknowledgment is made to Prof. F. P. McKibben and other members of the faculty for the facilities provided and for their interest in the work.

The pull-out tests of stirrups discussed in Section IV were made in the testing laboratory at the University of Pennsylvania in October, 1919, under the direction of S. C. Hollister of the Emergency Fleet Corporation. The facilities of the laboratory were made available for this investigation by arrangement with Prof. H. C. Berry.

Acknowledgment is made to A. S. Merrill, consulting concrete engineer of the Bureau of Standards, who has reviewed this paper. As a result of Mr. Merrill's work many portions of the paper have been modified, and some portions have been entirely reconstructed.

II. PULL-OUT TESTS WITH BARS TREATED TO PREVENT CORROSION

1. PRELIMINARY

This group of tests was made primarily to study the effect of the presence of anticorrosive coatings upon the bond resistance of the bars. A very complete series of tests made by Abrams[3] on uncoated bars was of much value in determining the type of specimen to be used, the technique of performing the tests, and the elimination of many undesirable variables which might have obscured the information sought.

The pull-out type of specimen used here has many advantages when it is desired to study a large number of variables. The specimen is easily and accurately made to any desired dimensions, can be tested quickly in a small testing machine, is fairly cheap, and gives results in usual terms with little calculation. For a comparative study like that reported here, it is an excellent type of specimen.

2. DESCRIPTION OF SPECIMEN

The pull-out specimen adopted for this work consisted of a one-half-inch steel reinforcing bar, 24 inches long, embedded axially in a concrete cylinder 6 inches high and 6 inches in diameter. In the position of molding and again in testing, the bar projected about one-half inch above the top of the cylinder and 17½ inches below the bottom.

In a total number of about 350 specimens, 18 kinds of protective coating were investigated. With a few exceptions, a set of 16 specimens was made with each kind of coating, and also with uncoated bars. In each set of specimens, 8 were made with plain square bars and 8 with deformed bars. Each group of 8 specimens was in turn divided into 4 pairs, which were stored under as many different conditions. Plain bars, except in series involving the use of metallic coatings, were painted by hand with a brush, while deformed bars were coated usually by dipping them into the paint. To supplement information obtained from these specimens, additional specimens were made in order to study the effect of varying the manner of application of certain paints.

[3] Bull. 71, Eng. Exp. Sta., Univ. of Illinois.

3. ANTICORROSIVE COATINGS

A list of the coatings used on the reinforcing bars in these tests is presented in Table 1. This table furnishes the key to the system of symbols used for distinguishing all of the test specimens. A complete specimen number indicates the series number of this investigation in the research program of the concrete ship section, the kind of coating, the kind of bar used and method of applying the coating, and the method of storage. For example, 8HA1 is the specimen number given to a pull-out specimen in series 8, in which an asphaltic coating, H, was applied to a plain square bar by means of a brush, after which the specimen 1 was stored in damp sand for 28 days before testing.

TABLE 1.—System of Reference Symbols Used to Indicate the Nature of the Coatings, Methods of Application, and Conditions of Storage

[Investigation series No. 8]

Kind of coating		Method of applying coating			Method of storage	
First reference letter	Composition	Second reference letter	Kind of bar	How applied	Final reference numeral	Conditions
A	No coating used.	A	Plain square	Painted with brush.	1 and 2	Damp sand, 28 days.
B	Coal-tar pitch, coal-tar naphtha.	B	Deformed square	Dipped.	3 and 4	Damp sand, 1 month, salt water, 5 months.
C	Asphalt paint.	C	do	Painted with brush.	5 and 6	Damp sand, 1 month; salt water and air, 5 months.
D	Do.	D	Plain square	Painted and sanded.	7 and 8	Damp sand, 1 month; air, 5 months.
E	Iron oxide, siliceous material, varnish, turpentine.	E	do	Dipped.		
F	Asphalt paint.					
G	Do.					
H	Do.					
I	Iron oxide, siliceous material, varnish, mineral spirits.					
J	Red lead, linseed oil dryer.					
K	Iron oxide, calcium sulphate, siliceous material, varnish, mineral spirits.					
L	Iron oxide, siliceous material, varnish, mineral spirits.					
M	Asphalt gilsonite paint.					
N	Coal-tar drying oil, petroleum distillate, turpentine.					
O	Zinc (galvanized).					
P	Zinc (sherardized).					
Q	Zinc (metal spray).					
V	Phosphate coating.					
X	Electrogalvanized.					

Table 2 indicates schematically the exact classification of all specimens tested.

TABLE 2.—Classification of Pull-Out Test Specimens

[The numerals indicate the number of specimens tested]

Reference letter	Coating	Storage																			
		Damp sand, 28 days Specimens 1 and 2					Damp sand, 1 month Salt water, 5 months Specimens 3 and 4					Damp sand, 1 month Salt water and air, 5 months Specimens 5 and 6					Damp sand, 1 month Air, 5 months Specimens 7 and 8				
	Kind	Plain painted	Deformed dipped	Deformed painted	Plain painted and sanded	Plain dipped	Plain painted	Deformed dipped	Deformed painted	Plain painted and sanded	Plain dipped	Plain painted	Deformed dipped	Deformed painted	Plain painted and sanded	Plain dipped	Plain painted	Deformed dipped	Deformed painted	Plain painted and sanded	Plain dipped
		A	B	C	D	E	A	B	C	D	E	A	B	C	D	E	A	B	C	D	E
A	(No coating used)	2	2				2	2				2	2				2	2			
B	Coal tar	2	2				2	2				2	2				2	2			
C	Asphaltic	2	2	2	2		2	2	2	2		2	2	2	2		2	2	2	2	
D	do	2	2				2	2				2	2				2	2			
E	Ferric oxide	2	2				2	2				2	2				2	2			
F	Asphaltic	2	2				2	2				2	2				2	2			
G	do	2	2				2	2				2	2				2	2			
H	do	2	2	2	2		2	2	2	2		2	2	2	2		2	2	2	2	
I	Ferric oxide	2	2				2	2				2	2				2	2			
J	Red lead	2	2				2	2				2	2				2	2			
K	Ferric oxide	2			2	2	2			2	2	2			2	2	2			2	2
L	do	2	2				2	2				2	2				2	2			
M	Asphaltic	2	2				2	2				2	2				2	2			
N	Coal tar	2	2		2	2	2	2				2	2		2	2	2	2		2	2
O	Galvanized	2	2				2	2									2	2			
P	Sherardized	2	2				2	2									1	1			
Q	Metal spray	2	2				2	2									2	2			
V	Phosphate film	2	2				2	2									2	2			
X	Electro galvanized	2					2					2					2				

4. MATERIALS

The aggregate used in making the specimens was a Potomac River sand, consisting principally of angular quartz grains and containing a slight amount of mica and clay. It was obtained in Arlington, Va. All of this sand passed a one-fourth-inch screen. A mechanical analysis made with a set of Howard & Morse sieves is presented in Table 3. The average tensile strength of three 1:3 cement mortar briquets was 250 pounds per square inch at seven days, which was 3 per cent higher than the average strength of similar briquets made with standard Ottawa sand.

TABLE 3.—Mechanical Analysis of Sand

Screen number	Separation size	Per cent passing	Screen number	Separation size	Per cent passing
	Inch			Inch	
¼-inch	0.250	100.0	50-mesh	0.011	24.3
10-mesh	.075	92.3	80-mesh	.0068	11.6
20-mesh	.034	76.1	100-mesh	.0055	7.5
30-mesh	.022	52.0	200-mesh	.0029	2.8
40-mesh	.015	35.2			

Tidewater Portland cement, having a fineness such that 83.9 per cent passed a No. 200 sieve, was used in making the concrete. In the concrete the proportions by weight were 1 part cement, 2 parts sand, and 0.485 parts water. With these proportions the water was 16.2 per cent (by weight) of all dry materials and 75 per cent (by volume) of the cement. Defined by sizes of aggregate, this mix was a mortar instead of a concrete, but, following the usage developed in connection with early concrete ship work, it is here termed concrete.

The steel used consisted of one-half-inch plain square bars and deformed bars of the same nominal cross section and perimeter.[4] The deformed bar was of the form shown in Fig. 1. It had nine lugs per foot of length. The lugs were truncated wedges about 0.05 inch in height, and included an angle of about 40° between the beveled bearing faces. It is evident that this form of lug gave some wedging action in the pull-out test, as verified by the fact that all pull-out specimens made with deformed bars failed by bursting the concrete block. No lateral reinforcement was used to restrain the concrete.

Physical properties of the steel used are given in Table 4.

TABLE 4.—Physical Properties of Steel

Type of bar	Yield point	Ultimate tensile strength	Ultimate elongation in 8 inches
	Lbs./in.²	Lbs./in.²	Per cent
½ inch, plain square	33 150	46 200	14.8
½ inch, deformed	61 300	105 750	16.6

In a few specimens the yield point of the bar was exceeded before failure occurred.

[4] A measurement of one plain bar and one deformed bar showed the perimeter of the deformed bar to be about 4 per cent less than that of the plain bar.

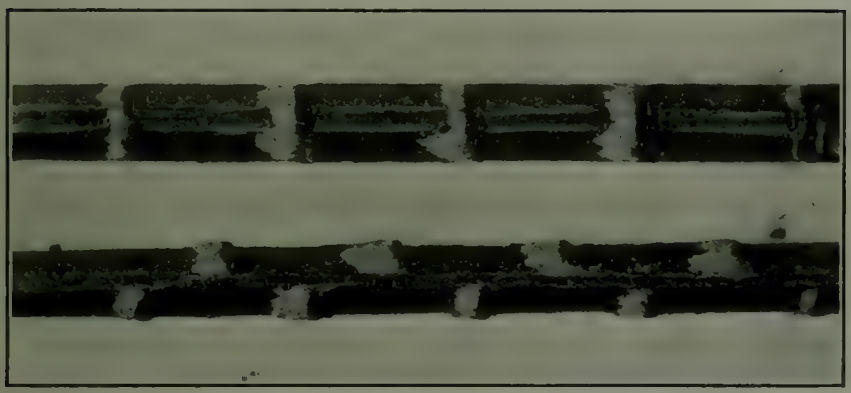


FIG. 1.—*Deformed bar used in pull-out tests*

5. MAKING OF SPECIMENS

The bars were prepared several days before the molding of the specimens. The plain bars were fairly smooth and had but little rust, and that was removed with emery cloth. One coat of paint was applied with a varnish brush over one-half of the length of each bar painted. The paint was allowed to dry for several days.

The deformed bars had a heavy coating of mill scale and rust. This coating was removed by scraping with the edge of a cold chisel and then brushing with a wire brush. Each bar was held vertically and the lower half of its length was dipped into the paint. The excess paint was allowed to drain off, and the bar was left to dry for several days. All paints were applied cold.

To compare the effect of different methods of applying the coating, a few specimens were made in which the plain bars were dipped and the deformed bars were painted with the brush. A few of the plain painted bars were also coated with a sharp fine sand immediately after painting, to study the effect of such a treatment.

Obviously, the above description does not refer to the application of metallic coatings, which were applied by commercial methods by the manufacturers to whom bars were furnished for such treatment.

In making the specimens, the cylinder molds were placed on a wooden bench. Holes (three-fourths inch in diameter) in the bench permitted the bars to be placed vertically in the axis of the cylinder, with the coated end projecting about one-half inch above the top of the mold. The bars were held in place in the bench by packing cotton waste tightly around them.

The concrete was tamped into place in the mold with a small wooden rod, and the top surface was smoothed off with a trowel.

6. AUXILIARY SPECIMENS

Three compression cylinders, 2 inches in diameter and 4 inches high, were made from the batches of concrete mixed for the pull-out test specimens. Average compressive strengths as determined for the different conditions of age and storage are as follows:

	Lbs./in.^{2}
Damp sand, 28 days	5090
Damp sand, 6 months	6195
Damp sand, 28 days, and sea water, 5 months	7010

7. STORAGE OF SPECIMENS

The specimens were removed from the molds when 24 hours old and were stored in damp sand in the laboratory for approximately one month. After this preliminary curing, different groups of specimens were accorded different treatment. In all cases specimens were allowed to stand in air 24 hours previous to testing.

Specimens 1 and 2 of each group were removed from the damp sand after 27 days and were tested on the following day. Specimens 3 and 4 were removed from the sand after 1 month and stored in artificial sea water for 5 months, then allowed to dry for 24 hours before testing.

Specimens 5 and 6 were removed from the sand when 1 month old and were stored in artificial sea water and in air alternately 12 hours each day for 5 months and then tested. These specimens were stored on a rack arranged to be readily lowered into and raised from the tank containing the sea water.

Specimens 7 and 8 were removed from the damp sand when 1 month old, stored in the laboratory uncovered for 5 months, and then tested.

All specimens were stored inside the laboratory where the range of temperatures was between 60 and 75° F. There is no indication that these temperatures produced harmful effects on any of the paint coatings.

The storage in sea water was to determine how the bond strength might be affected by the deterioration of paint and metallic coatings and by corrosion of the bars.

The artificial sea water referred to was prepared from the formula given in Table 5, based upon a typical chemical analysis of ocean water.

TABLE 5.—Composition of Artificial Sea Water

Chemical name	Symbol	Percentage composition of solids	Parts by weight
Potassium bromid	KBr	0.29	1
Potassium sulphate	K_2SO_4	2.55	9
Calcium sulphate	$CaSO_4$	3.97	14
Magnesium sulphate	$MgSO_4$	4.12	[a]14.5
Magnesium chlorid	$MgCl_2$	10.78	38
Magnesium carbonate	$MgCO_3$	.29	1
Sodium chlorid	NaCl	78.30	275
Water (spring)	H_2O	.....	10 000

[a] Anhydrous: equivalent to 29.7 parts of commercial magnesium and sulphate, $MgSO_4.7H_2O$.

8. METHOD OF TESTING

In making a pull-out test, the specimen was placed on the upper head of the testing machine, as shown in Fig. 2, with the long end of the bar passing down through a central hole in the spherical bearing block. The long end of the bar was engaged in the wedge grips in the moving head of the machine. In a number of cases readings of load were lost, due to the slipping of the bar in the

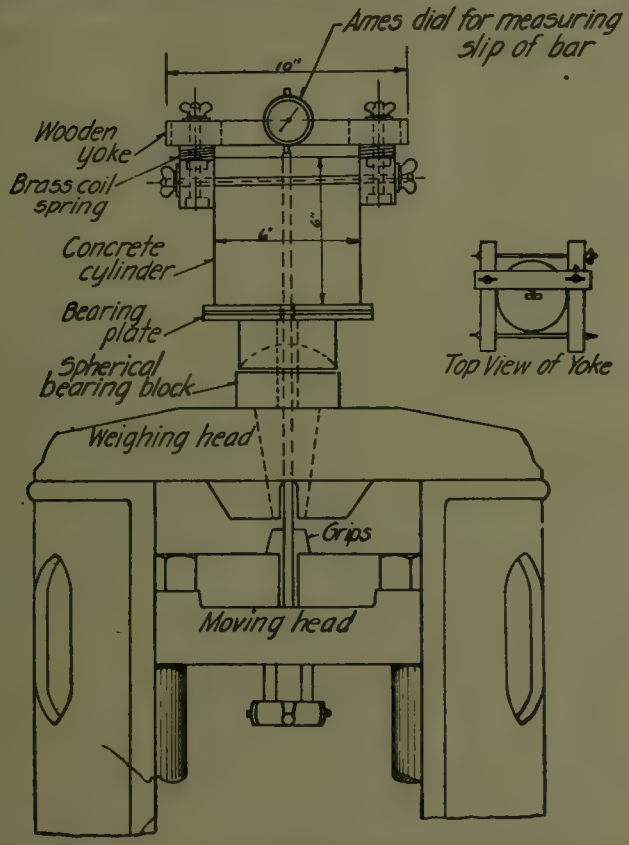


FIG. 2.—*Pull-out specimen in machine in position for testing*

grips at low loads, and in all cases this slipping made it difficult to keep the weighing beam of the machine in balance.[5]

The concrete block was embedded in plaster of Paris on a bearing plate, which transmitted the stress through a spherical bearing block to the weighing head of the machine. The plaster of Paris served to give a uniform bearing between the specimen and the

[5] Since it is particularly desirable to obtain accurate readings of load at the beginning of the slip of the bar, this test could probably be improved by providing a better connection between the bar and pulling head. This could be accomplished by threading the end of the bar and holding it with a nut against a plate bearing against the lower side of the pulling head.

bearing plate, and the spherical bearing allowed the bar to be adjusted in a vertical position, thus preventing the bending of the bar which otherwise would have occurred when the axis of the bar was not normal to the bearing surface of the concrete block.

The slip of the bar was measured at the upper end of the embedded bar by means of an Ames gage; this was supported by a wooden yoke clamped to the upper part of the concrete block in such a position that the plunger rested on the end of the bar. The dial was graduated so that its smallest division indicated a movement of 0.0001 inch, and a movement of 0.00005 inch was easily estimated. The latter was the smallest slip recorded. Load was applied continuously with the weighing beam kept carefully balanced, and as predetermined amounts of slip were reached, the corresponding load was read and recorded.

The tests were made on a power-driven Olsen testing machine of 20 000 pounds capacity. The slowest speed of the machine was used, giving the pulling head a movement of about 0.07 inch per minute. Due to the slipping of the bar in the grips at low loads, the stretching of the bar, and the compression in the plaster bearing, the slipping, as measured by the Ames gage, was much less than the movement of the pulling head.

9. NATURE OF THE BOND RESISTANCE OF A COATED BAR

A study of bond between concrete and uncoated steel has indicated that bond resistance is made up of two parts, adhesive resistance and sliding resistance. Adhesive resistance comes into play before the bar begins to slip. Sliding resistance is evidently due to friction between the concrete and the surface of the reinforcing bar, which may be, comparatively speaking, rough and irregular in form.

With the deformed bar the projecting lugs provide additional resistance after slipping has begun. It is believed that the adhesive resistance between the concrete and the longitudinal surfaces of the bar is destroyed before there is enough movement to develop much compressive stress between the beveled surface of the lug and the concrete in contact with it.

The action of a coated bar embedded in concrete is undoubtedly of a different nature from that of an uncoated bar. With the more plastic coatings especially, slipping may be due to three possible causes, as follows: (1) Slip between the coating and the concrete; (2) flowing or shearing deformation in the coating in the direction of motion; and (3) slip between the coating and the bar.

A very soft paint may act merely as a lubricant tending to facilitate slipping of the bar, while a hard, brittle paint may crumble when acted upon by shearing forces. Although the resistance of these paints to distortion is not known, it seems likely that the shearing strength of a paint is less than its adhesion to steel or concrete. On the other hand, a metallic coating, such as zinc, is elastic and has a comparatively high shearing strength, so that failure might be expected to occur through slipping of the zinc on the concrete. A study of the tests shows a considerable variation in the behavior of the different coatings.

10. GENERAL CHARACTERISTICS OF RESULTS

The bond-slip data of all tests have been plotted in Figs. 4 to 38, inclusive, which show the relation between bond stress and slip of bar. Each figure contains the information secured with the use of a certain coating.

The values of bond stress for end slips up to 0.01 inch have been plotted on an extended scale in the left-hand portion of each diagram in order to show distinctly the zone in which end-slip values began to increase rapidly; the same data are included in the right-hand portion of each diagram, which shows values of bond stress for end slips up to 0.20 inch. The points plotted in each diagram represent the average results of tests of two similar specimens. The different conditions of storage have been indicated by the same conventions throughout the diagrams.

The form of the bond-slip curves for coated bars is, in general, similar to that for uncoated bars. A few outstanding differences may be noted briefly. While the plain uncoated bar reached its maximum bond resistance at a slip of 0.01 inch or less, after which the bond stress decreased with further slip, many of the plain painted bars did not reach maximum bond resistance until a slip of 0.03 to 0.05 inch had occurred, and the bond stress remained nearly constant with considerable increase in slip.

However, with some metallic coatings the maximum bond resistance on plain bars was developed at a slip of 0.001 inch and was followed by a sudden large slip of bar with a correspondingly large reduction in bond stress.

Uncoated deformed bars showed an increase in bond resistance with an increase in slip throughout the test. The results for coated deformed bars show two noticeable features: (1) The bond stress was low for small amounts of slip, but the general slope of the bond-slip curves is about the same as for the uncoated bars; (2) in some cases the bond stress developed at the initial slip

remained constant, or even decreased, until a slip of 0.02 to 0.03 inch was reached, after which the bond-slip curves took on the characteristic slope of the curve for uncoated bars. It is evident that the point at which a curve takes this sudden change in slope corresponds to the slip at which the lugs of the deformed bar have reached a firm bearing against the adjoining concrete.

Tests reported elsewhere[6] have indicated that a very small amount of slip corresponds to critical bond stress in a reinforced concrete beam. For this reason comparisons have been made of bond stresses corresponding to a slip of 0.001 inch and 0.01 inch, respectively. The ratios of these stresses to the maximum bond stresses developed in these tests are given in Table 6 in the form of percentages.

TABLE 6.—Bond Stresses at Specified Slips in Percentage of Maximum Bond Stresses

[Values given are averages for all ages and conditions of storage]

Reference letter	Coating: Kind	0.001-inch slip: Plain bars	0.001-inch slip: Deformed bars	0.01-inch slip: Plain bars	0.01-inch slip: Deformed bars
		Per cent	Per cent	Per cent	Per cent
A	(No coating used)	94	53	[illegible]	62
C	Asphaltic	58	[illegible]	[illegible]	13
D	Asphaltic	60	23	90	36
F	Asphaltic	55	15	79	22
G	Asphaltic	53	23	86	[illegible]
H	Asphaltic	74	33	[illegible]	44
M	Asphaltic	62	26	92	45
	Asphaltic, average	60	21	[illegible]	32
B	Coal tar	65	[illegible]	[illegible]	34
N	Coal tar	68	26	98	37
	Coal tar, average	67	23	93	36
E	Ferric oxide	67	24	[illegible]	[illegible]
I	Ferric oxide	84	33	97	[illegible]
K	Ferric oxide	[illegible]	.....	[illegible]	.....
L	Ferric oxide	73	[illegible]	95	41
	Ferric oxide, average	73	29	90	41
O	Metallic	[illegible]	50	80	44
P	Metallic	100	[illegible]	53	45
R	Metallic	101	.....	74	.....
	Metallic, average	100	[illegible]	69	45
Q	Metal spray	16	4	76	[illegible]
J	Red lead	47	7	66	15
V	Phosphate	92	[illegible]	97	[illegible]

[6] Abrams, Bull. 71, Eng. Exp. Sta., Univ. of Illinois.

In order to present graphically a large amount of the information of these tests, Fig. 3 has been prepared. Bond stresses at slips of 0.001 inch and 0.01 inch, and at maximum load are shown. The chart shows that, in general, the bond resistance of coated bars was much less than that for uncoated bars. The dotted horizontal lines drawn in Fig. 3, corresponding to the bond stresses developed in uncoated bars at a slip of 0.001 inch and at the maximum load, enable this comparison to be made readily. Averaging the results for all conditions of storage for each coating, the data of Fig. 3 are summarized and shown as percentages in Table 7.

TABLE 7.—Bond Stresses for Coated Bars in Percentage of Bond Stresses for Uncoated Bars

[Values given are averages for all ages and conditions of storage]

Coating		Percentage of bond stress for uncoated bars						
Reference letter	Kind	0.001-inch slip		0.01-inch slip		Maximum bond stress		Average
		Plain	Deformed	Plain	Deformed	Plain	Deformed	
A	Uncoated	100	100	100	100	100	100	100
C	Asphaltic	10	9	43	11	47	69	
D		29	29	41	37	45	65	
F		30	19	40	23	50	67	
G		21	21	13	36	45	61	
H		38	45	46	50	48	71	
M		41	50	66	68	71	85	
	Average	34	27	46	35	53	67	43
B	Coal tar	53	25	67	33	76	67	
N		52	38	83	47	84	78	
	Average	53	32	75	42	80	73	60
E	Ferric oxide	30	25	35	30	42	80	
I		45	36	48	40	71	56	
K		37	.....	43	.....	52	.....	
L		40	31	33	47	59	71	
	Average	38	16	34	45	43	69	47
O	Metallic	76	71	58	59	72	82	
P		89	94	12	50	80	76	
X		97	.....	66	.....	89	.....	
	Average	87	86	52	54	80	79	75
Q	Metal spray	1	2	5	17	7	88	21
J	Red lead	6	9	8	15	8	63	18
V	Phosphate	90	107	88	104	91	116	99

11. ACTION OF INDIVIDUAL COATINGS

A brief tabular summary of the characteristic action of the various coatings is given in Table 8. As an additional guide in determining the merits of a given type of coating, the resistance of each coating to salt-spray corrosion tests is also presented.

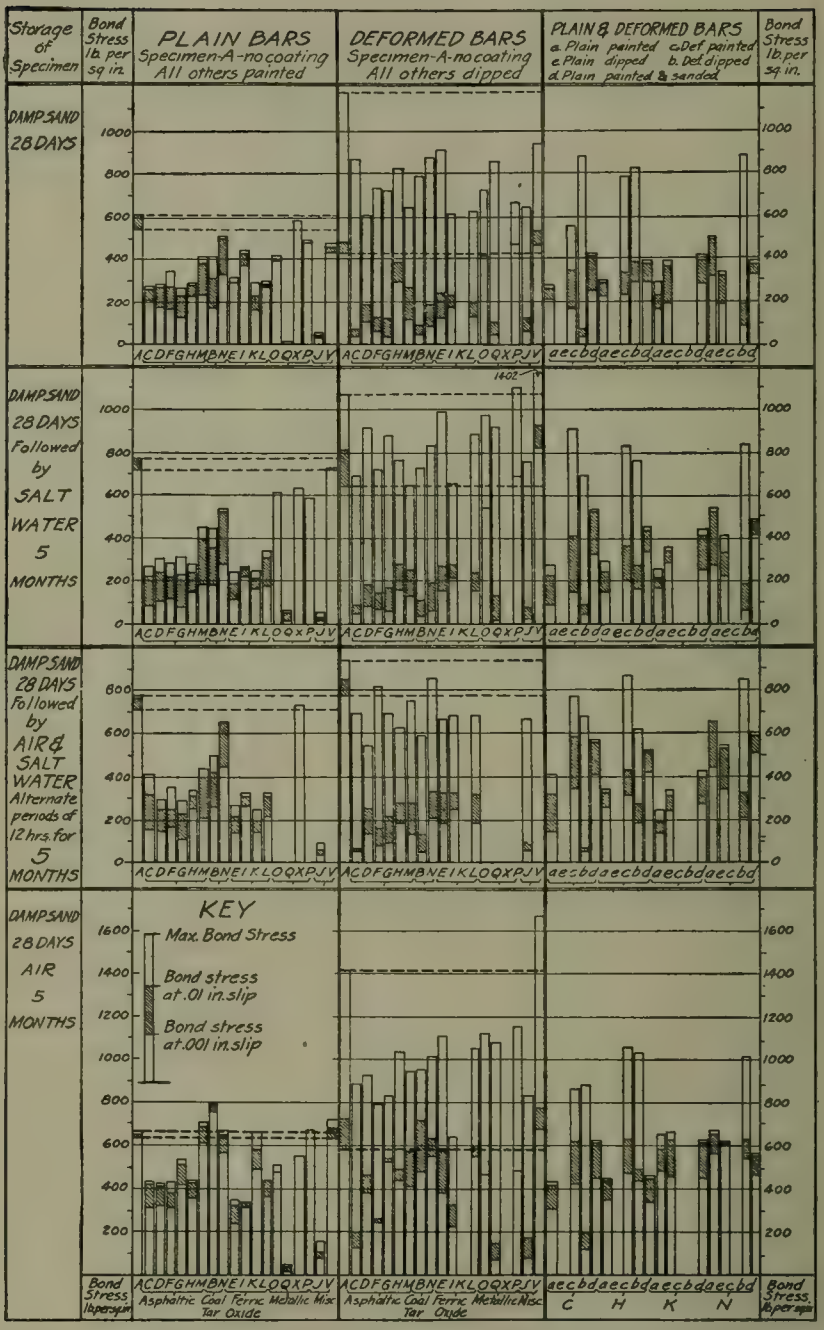

Storage of Specimen
Bond Stress lb. per sq. in.
PLAIN BARS
Specimen-A-no coating
All others painted
DEFORMED BARS
Specimen-A-no coating
All others dipped
PLAIN & DEFORMED BARS
a. Plain painted c. Def. painted
e. Plain dipped b. Def. dipped
d. Plain painted & sanded
DAMP SAND 28 DAYS
DAMP SAND 28 DAYS Followed by SALT WATER 5 MONTHS
1402
DAMP SAND 28 DAYS Followed by AIR & SALT WATER Alternate periods of 12 hrs. for 5 MONTHS
DAMP SAND 28 DAYS AIR 5 MONTHS
KEY
Max. Bond Stress
Bond stress at .01 in. slip
Bond stress at .001 in. slip
A C D F G H M B N E I K L O Q X P J V
a e c b d a e c b d a e c b d a e c b d
Asphaltic Coal Ferric Metallic Misc. Tar Oxide
C H K N

FIG. 3.—*Bond stresses developed with different coatings under various conditions of storage*

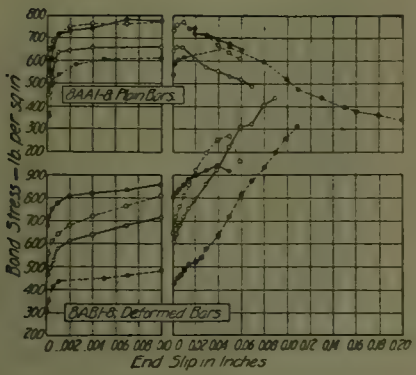


FIG. 4.—*Bond-slip diagrams for uncoated bars*

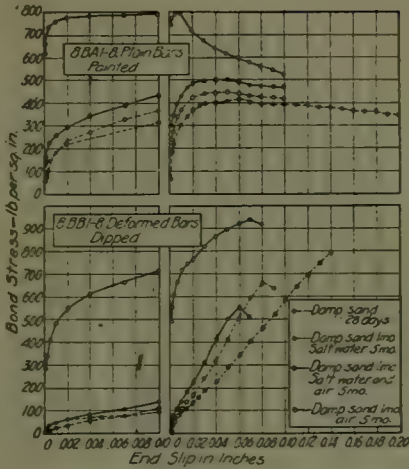

8BA1-8. Plain Bars Painted
8BB1-8. Deformed Bars Dipped
Damp sand 28 days
Damp sand 1mo. Salt water 5mo.
Damp sand 1mo. Salt water and air 5mo.
Damp sand 1mo. air 5mo.
Bond Stress-lb per sq in.
End Slip in Inches

FIG. 5.—*Bond-slip diagrams for coating B; coal tar*

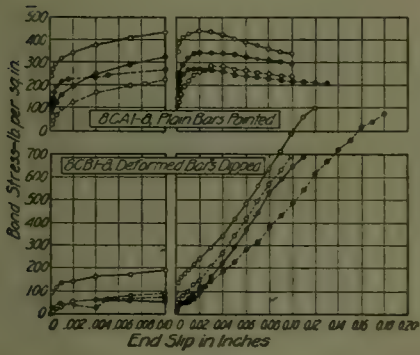


FIG. 6.—*Bond-slip diagrams for coating C; asphaltic*

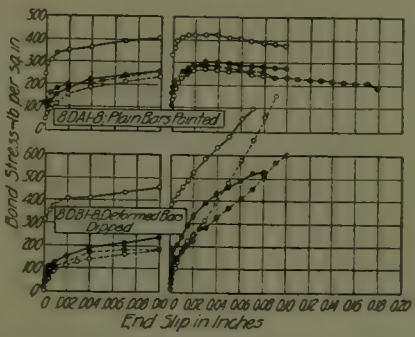


FIG. 7.—*Bond-slip diagrams for coating D; asphaltic*

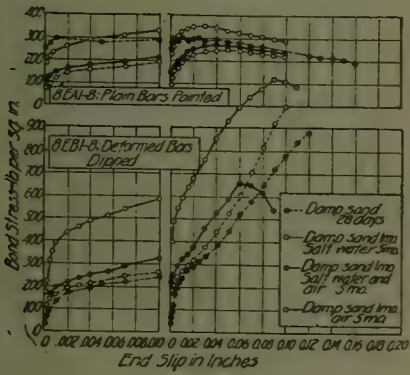

8EA1-8. Plain Bars Painted
8EB1-8. Deformed Bars Dipped
Damp sand 28 days
Damp sand 1mo. Salt water 5mo.
Damp sand 1mo. Salt water and air 5mo.
Damp sand 1mo. air 5mo.
Bond Stress-lb per sq in.
End Slip in Inches

FIG. 8.—*Bond-slip diagrams for coating E; ferric oxide*

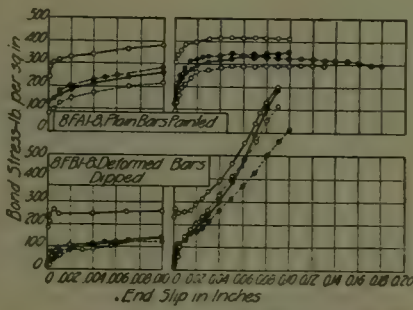


FIG. 9.—*Bond-slip diagrams for coating F; coal tar*

NOTE FOR FIGS. 4 TO 38, INCLUSIVE.—In the left-hand section of each figure the slip, up to 0.01 inch, is plotted horizontally to a more extended scale than that which is used in the right-hand section.

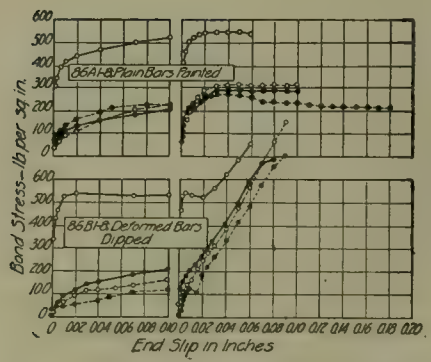


FIG. 10.—*Bond-slip diagrams for coating G; asphaltic*

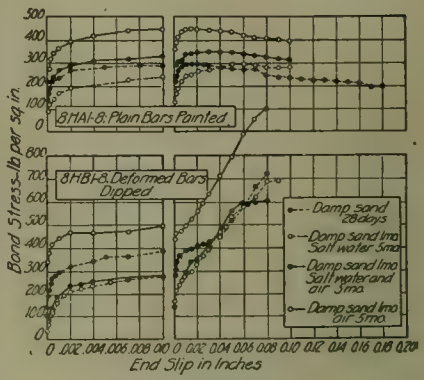

8HA1-8. Plain Bars Painted
8HB1-8. Deformed Bars Dipped
Damp Sand 28 days
Damp sand 1mo Salt water 5mo
Damp sand 1mo Salt water and air 3mo
Damp sand 1mo air 3mo
Bond Stress-lb per sq in.
End Slip in Inches

FIG. 11.—*Bond-slip diagrams for coating H; asphaltic*

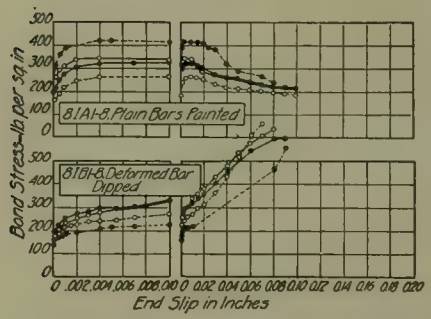


FIG. 12.—*Bond-slip diagrams for coating I; ferric oxide*

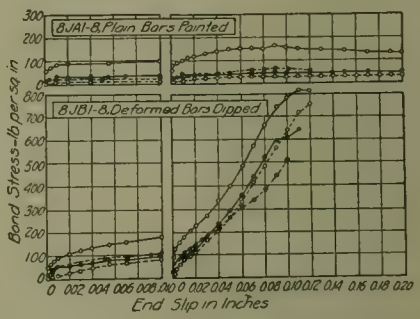


FIG. 13.—*Bond-slip diagrams for coating J; red lead*

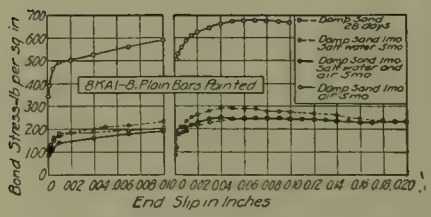

8KA1-8. Plain Bars Painted
Damp Sand 28 days
Damp Sand 1mo Salt water 5mo
Damp Sand 1mo Salt water and air 3mo
Damp Sand 1mo air 3mo
Bond Stress-lb per sq in.
End Slip in Inches

FIG. 14.—*Bond-slip diagrams for coating K; ferric oxide*

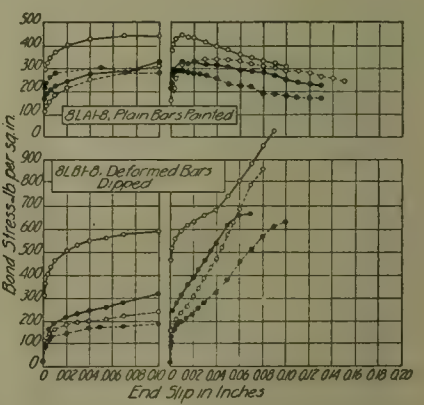


FIG. 15.—*Bond-slip diagrams for coating L; ferric oxide*

NOTE FOR FIGS. 4 to 38, INCLUSIVE.—In the left-hand section of each figure the slip, up to 0.01 inch, is plotted horizontally to a more extended scale than that which is used in the right-hand section.

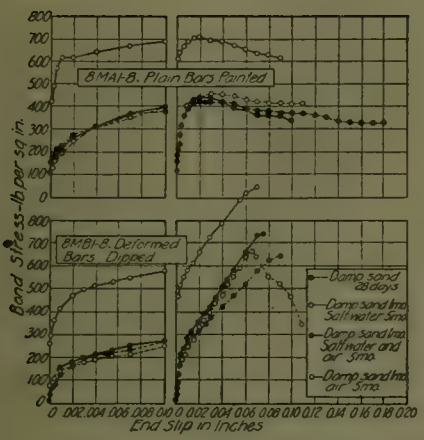

8MA1-8. Plain Bars Painted
8MB1-8. Deformed Bars Dipped
Damp sand 28 days
Damp sand 1mo Salt water 5mo
Damp sand 1mo Salt water and air 5mo
Damp sand 1mo air 5mo
Bond Stress-lb per sq in.
End Slip in Inches

FIG. 16.—*Bond-slip diagrams for coating M; asphaltic*

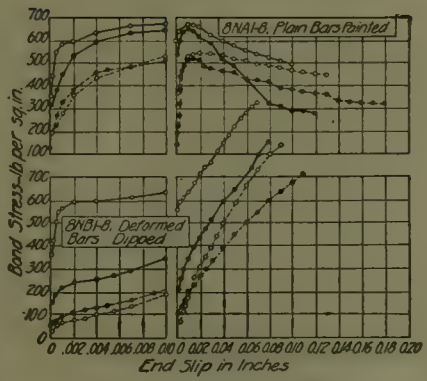


FIG. 17.—*Bond-slip diagrams for coating N; coal tar*

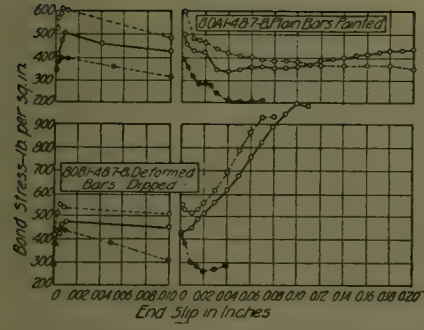


FIG. 18.—*Bond-slip diagrams for coating O; galvanized*

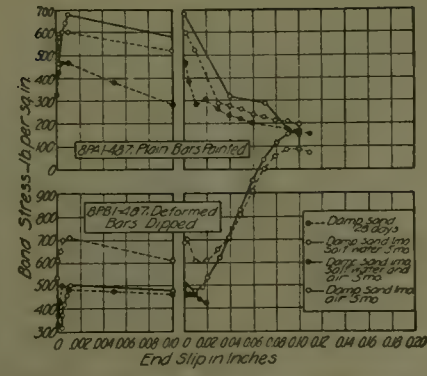

8PA1-4&7. Plain Bars Painted
8PB1-4&7. Deformed Bars Dipped
Damp sand 28 days
Damp sand 1mo Salt water 5mo
Damp sand 1mo Salt water and air 5mo
Damp sand 1mo air 5mo
Bond Stress-lb per sq in.
End Slip in Inches

FIG. 19.—*Bond-slip diagrams for coating P; sherardized*

NOTE FOR FIGS. 4 to 38, INCLUSIVE.—In the left-hand section of each figure the slip, up to 0.01 inch, is plotted horizontally to a more extended scale than that which is used in the right-hand section.

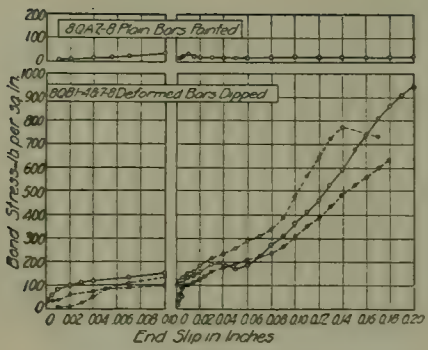


FIG. 20.—*Bond-slip diagrams for coating Q; metal spray*

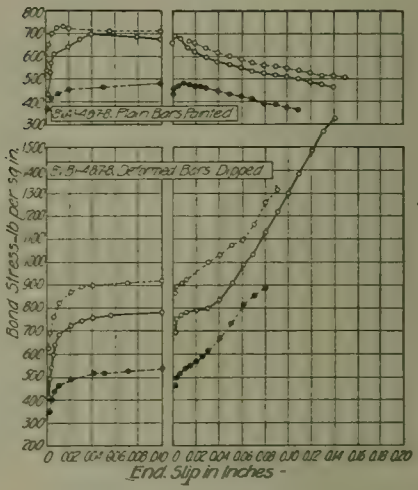


FIG. 21.—*Bond-slip diagrams for coating V; phosphate film*

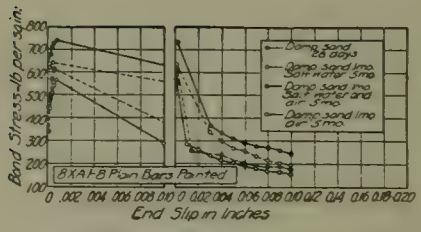

Damp sand 28 days
Damp sand 1mo Salt water 5mo
Damp sand 1mo Salt water and air 5mo
Damp sand 1mo air 5mo
8XA1-8 Plain Bars Painted
Bond Stress-lb per sq in.
End Slip in Inches

FIG. 22.—*Bond-slip diagrams for coating X; electro galvanized*

NOTE FOR FIGS. 4 to 38, INCLUSIVE.—In the left-hand section of each figure the slip, up to 0.01 inch, is plotted horizontally to a more extended scale than that which is used in the right-hand section.

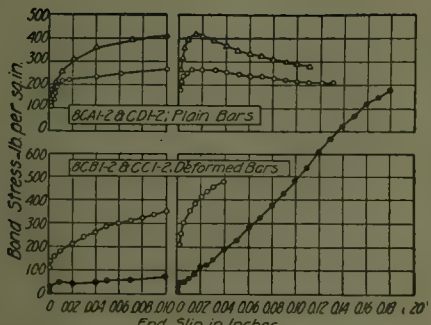


FIG. 23.—*Coating C, stored in damp sand 28 days*

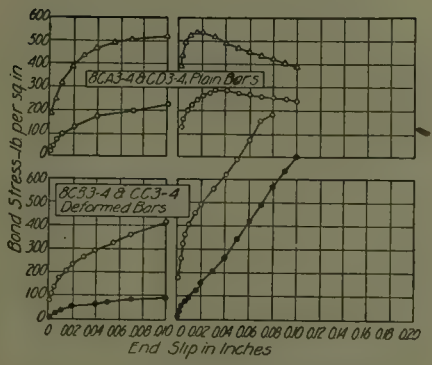


FIG. 24.—*Coating C, stored in damp sand one month, salt water five months*

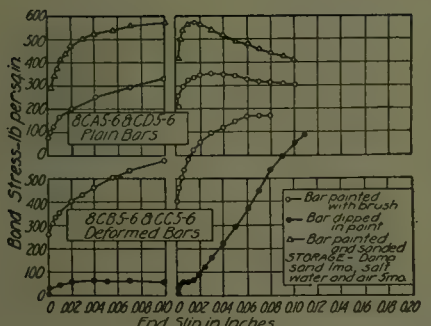

Bond Stress-lb per sq.in.
8CA5-6 & CD5-6 Plain Bars
8CB5-6 & CC5-6 Deformed Bars
Bar painted with brush
Bar dipped in paint
Bar painted and sanded
STORAGE - Damp Sand 1mo, Salt water and air 3mo.
End Slip in Inches

FIG. 25.—*Coating C, stored in damp sand one month, salt water and air five months*

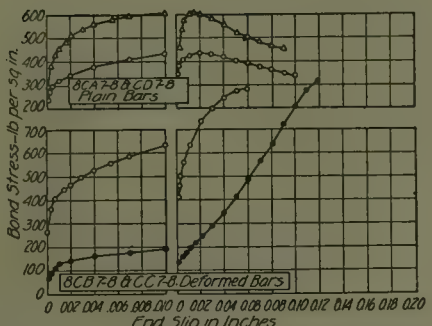


FIG. 26.—*Coating C, stored in damp sand one month, air five months*

NOTE FOR FIGS. 4 TO 38, INCLUSIVE.—In the left-hand section of each figure the slip, up to 0.01 inch, is plotted horizontally to a more extended scale than that which is used in the right-hand section.

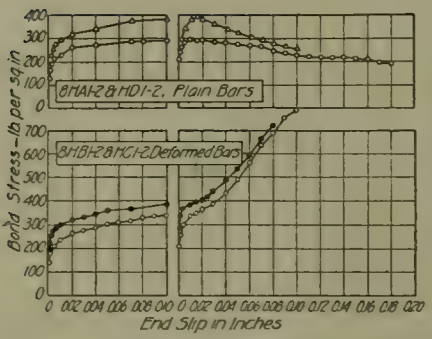


FIG. 27.—*Coating H, stored in damp sand 28 days*

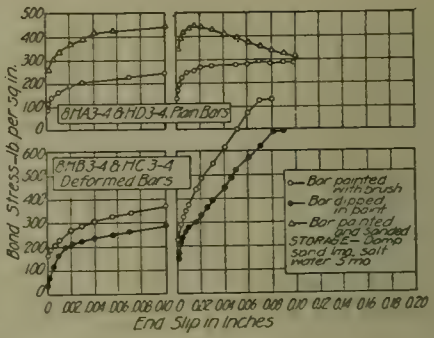

Bond Stress-lb per sq in
End Slip in Inches
8 HA3-4 & HD3-4, Plain Bars
8 HB3-4 & HC3-4 Deformed Bars
Bar painted with brush
Bar dipped in paint
Bar painted and sanded
STORAGE—Damp sand 1 mo, salt water 5 mo

FIG. 28.—*Coating H, stored in damp sand one month, salt water five months*

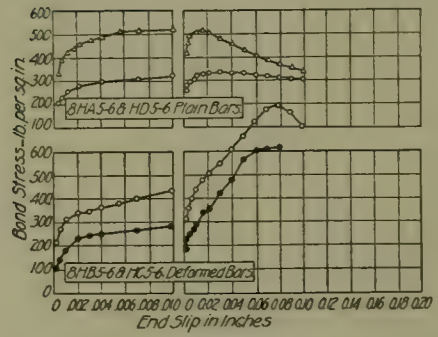


FIG. 29.—*Coating H, stored in damp sand one month, salt water and air five months*

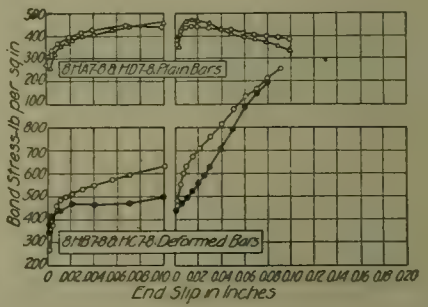


FIG. 30.—*Coating H, stored in damp sand one month, air five months*

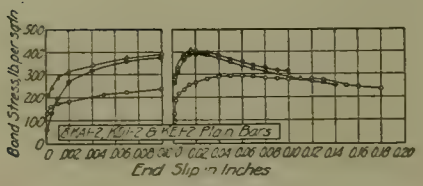


FIG. 31.—*Coating K, stored in damp sand 28 days*

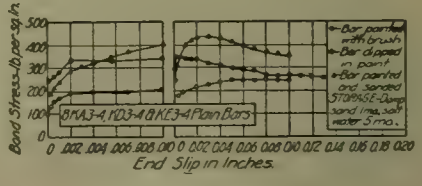

Bond Stress-lb per sq in
End Slip in Inches
8 HA3-4, KD3-4 & KE3-4 Plain Bars
Bar painted with brush
Bar dipped in paint
Bar painted and sanded
STORAGE—Damp sand 1 mo, salt water 5 mo.

FIG. 32.—*Coating K, stored in damp sand one month, salt water five months*

NOTE FOR FIGS. 4 TO 38, INCLUSIVE.—In the left-hand section of each figure the slip, up to 0.01 inch, is plotted horizontally to a more extended scale than that which is used in the right-hand section.

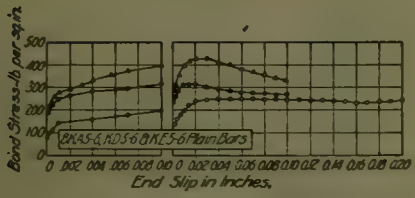


FIG. 33.—*Coating K, stored in damp sand one month, salt water and air five months*

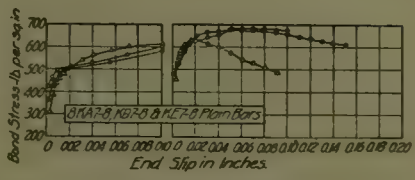


FIG. 34.—*Coating K, stored in damp sand one month, air five months*

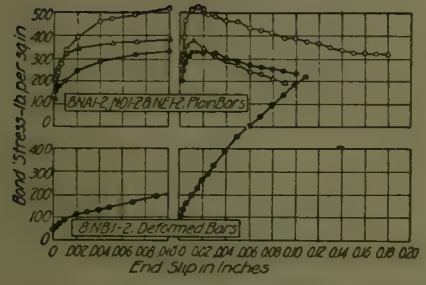


FIG. 35.—*Coating N, stored in damp sand 28 days*

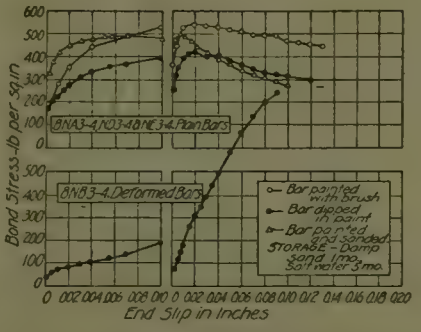

Bond Stress-lb per sq in
8 NA3-4, ND3-4 & NE3-4 Plain Bars
8 NB3-4, Deformed Bars
Bar painted with brush
Bar dipped in paint
Bar painted and sanded
STORAGE - Damp Sand 1 mo. Salt water 3 mo.
End Slip in Inches

FIG. 36.—*Coating N, stored in damp sand one month, salt water five months*

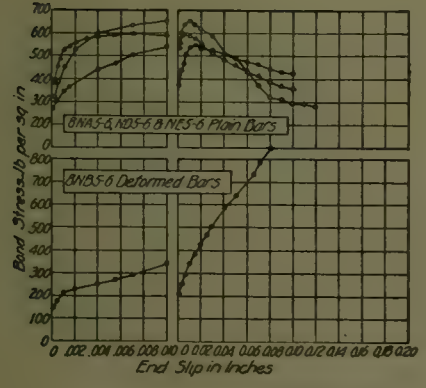


FIG. 37.—*Coating N, stored in damp sand one month, salt water and air five months*

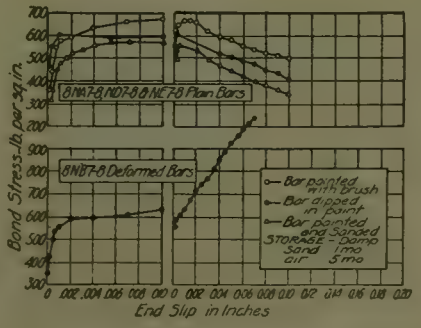

Bond Stress-lb per sq in
8 NA7-8, ND7-8 & NE7-8 Plain Bars
8 NB7-8 Deformed Bars
Bar painted with brush
Bar dipped in paint
Bar painted and sanded
STORAGE - Damp Sand 1 mo. air 5 mo.
End Slip in Inches

FIG. 38.—*Coating N, stored in damp sand one month, air five months*

NOTE FOR FIGS. 4 TO 38, INCLUSIVE.—In the left-hand section of each figure the slip, up to 0.01 inch, is plotted horizontally to a more extended scale than that which is used in the right-hand section.

TABLE 8.—Characteristic Action of Coatings in Bond Tests and Corrosion Tests

Reference letter	Composition and appearance of coating	Average time of drying		Ratio of bond stress for coated bar to uncoated bar (0.01-inch slip)		Characteristic feature of bond tests	Resistance to salt-spray corrosion test
		Plain	Deformed	Plain	Deformed		
		Days	Days				
B	Coal tar; dried to dull finish, with tough adhesive film.	2	5	0.67	0.36	Bond stress high after 6 months' storage in air; fairly low after other storage; above average on plain bars.	Failed.
C	Asphaltic; surface sticky and not very adhesive.	3	6	.43	.14	Comparatively low, especially on deformed bars; below average of all coatings.	Fair.
D	Asphaltic; fairly hard and tough surface.	3	5	.41	.37	Comparatively low; below average of all coatings.	Do.
E	Ferric oxide; adhesive surface.	2	4	.36	.49	Better result on deformed than on plain bars; about average of all coatings; shows well on 28-day test.	Good.
F	Coal tar; dried to dull finish with tough adhesive film.	2	5	.40	.23	Bond stress high after 6 months' storage in air; fairly low after other storage; above average on plain bars.	Fair.
G	Asphaltic; slightly sticky, glossy and adhesive.	3	6	.42	.36	Slightly below average of all coatings; best after 6 months' storage in air; maximum bond of plain bars continues with considerable slip.	Do.
H	Asphaltic; film sticky and glossy, not very adhesive.	2	6	.46	.50	Slightly above average; paint was scaly after specimen was broken and adhered partly to concrete.	Do.
I	Ferric oxide; hard, dull, rather rough finish.	3	4	.48	.40	Average of all coatings; adhered to concrete of broken specimens; form of bond-slip curves similar to those of uncoated bars.	Good.
J	Red lead; thick, wrinkled film; dried very slowly.	.....	.....	.08	.16	Very poor bond; fair maximum bond on deformed bars; paint adhered to both steel and concrete of broken specimen; appeared chalky.	Excellent.
K	Ferric oxide; smooth, glossy film.	.....	.....	.43	.....	Slightly below average; fair after 6 months' storage in air; maximum bond continued with large slips.	Do.

TABLE 8—Continued

Reference letter	Composition and appearance of coating	Average time of drying		Ratio of bond stress for coated bar to uncoated bar (0.01-inch slip)		Characteristic feature of bond tests	Resistance to salt-spray corrosion test.
		Plain	Deformed	Plain	Deformed		
		Days	Days				
L	Ferric oxide; paint thick and poor consistency.	5	[illegible]	0.48	0.47	Slightly above average; best results with 6 months' storage in air; adhered mainly to concrete in broken specimens.	Failed.
M	Asphaltic; hard, glossy film.	[illegible]	4	.66	.48	Better than average; paint appeared slightly brittle in broken specimens.	Fair.
N	Coal tar; film lumpy, not of uniform thickness.	7	[illegible]	.83	.47	Considerably above average; bond-slip curves similar to those for uncoated bars; paint adhered to broken specimens.	Failed.
O	Zinc (galvanized); film about 0.003 inch thick.	.....	.....	.58	.59	Much above average; maximum bond at 0.001-inch slip on plain bars followed by sudden slip and decrease of bond; peculiar bond-slip curves for deformed bars.	
P	Zinc (sherardized); film about 0.002 inch thick.	.....	.....	.42	.69	Much above average; maximum bond on plain bars at 0.001-inch slip; action similar to "O."	
Q	Zinc (metal spray); film about 0.001 inch thick.	.....	.....	.05	.17	Very poor bond; a fair maximum bond is developed with deformed bars; practically no bond with plain bars.	
V	Phosphate.....	.....	.....	.88	1.04	Very good bond; action similar to that of uncoated bars; consistently good at all storages.	
X	Zinc (electro galvanized)	.....	.....	.66	.....	Good bond, very much above average; behavior similar to that of coatings "O" and "P."	

12. COMPARISON OF PLAIN AND DEFORMED BARS

The relation between bond stresses developed by plain bars and those developed by deformed bars has already been shown in the bond-slip curves and in Fig. 3. In order to provide a definite numerical comparison, the information of Table 9 was prepared. Results given for coated bars are the average for all coatings tested.

TABLE 9.—Comparison of Bond Stresses in Plain and Deformed Bars

[Values given are averages for all ages and all conditions of storage]

Treatment of bar	Bond stress at 0.001-inch slip		Ratio, plain to deformed	Bond stress at 0.01-inch slip		Ratio, plain to deformed	Maximum bond stress		Ratio, plain to deformed
	Plain	Deformed		Plain	Deformed		Plain	Deformed	
	Lbs/in.²	Lbs/in.²		Lbs/in.²	Lbs/in.²		Lbs/in.²	Lbs/in.²	
Uncoated	655	610	1.07	704	715	0.99	[illegible]	1153	0.62
Coated	[illegible]	220	1.31	348	293	1.19	[illegible]	836	.48

Conclusions based on the relation of bond stresses for plain and deformed bars, as shown in Table 9, must be qualified by the fact that the method of applying the coatings was different for the plain bars from that used for the deformed bars. This subject is discussed in Section II–14.

13. EFFECT OF VARIATIONS IN CONDITIONS OF STORAGE

Fig. 3 shows in a general way that the bond strength for specimens six months old was considerably higher for those stored in air than for the corresponding specimens stored under other conditions. In order to facilitate comparison, Table 10 has been prepared. This table shows the effect on the bond strength of the various conditions of storage.

With plain bars continuous storage in sea water produced about the same effect as alternate storage in sea water and air. Bond stresses from both of these averaged about three-fourths as high as those developed with the specimens stored in air. The effect of a given method of storage does not show consistent results for the plain and deformed types of bar, but in general no appreciable difference in bond stress may be expected between the two conditions of continuous and intermittent exposure to sea water.

The unprotected ends of bars were much more badly corroded in the case of intermittent exposure to sea water than in the case

of continuous exposure, but examination of the specimens showed that this condition did not extend to the embedded portion of the bars.

TABLE 10.—Effect of Conditions of Storage on Bond Strength

[Maximum bond stress for various methods of storage in percentage of maximum bond stress for storage in damp sand 28 days and in air 5 months]

Coating		Plain bars			Deformed bars		
Reference letter	Kind	Damp sand 28 days	Damp sand 28 days Sea water 5 months	Damp sand 28 days Air and sea water 5 months	Damp sand 28 days	Damp sand 28 days Sea water 5 months	Damp sand 28 days Air and sea water 5 months
		Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
A	Uncoated	92	116	115	82	75	66
C	Asphaltic	84	61	79	97	78	77
D		67	54	69	65	95	58
F		80	68	83	92	91	101
G		50	58	54	87	103	80
E		83	63	77	79	74	59
M		89	64	72	68	69	78
	Average	64	63	71	81	85	76
B	Coal tar	51	57	64	83	76	61
N		76	81	97	87	82	84
	Average	63	69	81	85	79	73
H	Ferric oxide	84	74	77	81	89	60
I		128	77	95	95	102	105
K		43	59	37	.....	.....	.....
L		68	78	75	59	84	54
	Average	81	80	71	78	92	76
O	Metallic	81	120	.....	88	97	.....
P		71	89	.....	43	95	.....
X		105	113	129	.....	.....	.....
	Average	86	107	129	83	91	.....
Q	Metal spray	.....	125	.....	72	84	.....
J	Red lead	35	88	56	77	92	80
V	Phosphate	67	103	.....	97	84	.....
	Average (all specimens)	71	78	78	76	86	75

14. EFFECT OF MANNER OF APPLYING COATINGS

In order to determine whether or not the bond strength of coated bars was affected by the manner of applying the coatings, specimens were made with four paints, C, H, K, and N (see Table 2), using three methods of application. These paints represented the asphaltic, ferric oxide, and coal-tar types. The bond-slip curves for this particular study are plotted in Figs. 23 to 38, and the

information is also shown in Fig. 3. The curves show some contradictory results, in that for cases which are comparable the highest bond resistance was sometimes obtained with painted bars and sometimes with dipped bars.

Table 11 shows relative values of bond stresses at 0.01 inch slip for all methods of applying coatings. For convenience, results from painted bars have been taken as unity. As four paints only were subjected to this comparison, the relations shown in this table should not be regarded as conclusive.

TABLE 11.—Relative Values of Bond Resistance at 0.01-Inch Slip—Effect of Various Methods of Applying Coating

Method of applying coating	"C" asphaltic		"H" asphaltic		"K" ferric oxide	"N" coal tar
	Plain bar	Deformed bar	Plain bar	Deformed bar	Plain bar	Plain bar
Painting	1.00	1.00	1.00	1.00	1.00	1.00
Dipping	.....	0.19	.....	0.84	1.46	0.70
Painting and sanding	1.74	.....	1.45	.....	1.65	.86

15. SUMMARY

Results of this series of tests have shown a wide variation in the behavior of different coatings. A few of the results may be summarized here for convenient reference.

(*a*) The maximum bond stress developed by bars which had been painted was generally considerably less than for unpainted bars, but the reduction in maximum bond stress due to galvanizing and some similar processes was less than that due to painting.

(*b*) With the exception of certain metallic coatings, the maximum bond stress for plain coated bars was reached after considerably greater slip than with uncoated bars. The amount of slip of coated bars at maximum stress was often 0.02 to 0.03 inch. Furthermore, the bond resistance did not decrease as rapidly as with uncoated bars with continued slip after the maximum load had been reached. Certain metallic coatings, however, reached maximum bond resistance with very small slip, followed by a sudden decrease in bond load with increasing slip. In a reinforced concrete beam under a constant load, such a yielding of the bond resistance might cause failure without warning.

(*c*) Coated deformed bars apparently slipped considerably before the corrugations or lugs reached a firm bearing. After this had taken place, the increase of resistance with increase of slip was similar to that in uncoated deformed bars.

(*d*) The bond resistance at a slip of 0.001 inch was a much smaller proportion of the maximum bond resistance for coated bars than for uncoated bars. A slip of 0.001 inch has been considered by some[7] to produce critical conditions of bond stress in a beam.

(*e*) For pull-out specimens employing coated bars, continuous and intermittent storage in artificial sea water resulted in bond strength averaging about three-fourths as great as from storage in air.

(*f*) Sanding of painted bars tended to increase the bond resistance in a number of cases; also dipping bars in paint often resulted in a lower maximum bond stress than was found for bars painted with a brush. However, the effectiveness of the manner in which a coating is applied seemed to depend upon the nature of the paint, and should be determined by test in every case in which it is important to have this information.

III. TESTS OF REINFORCED CONCRETE BEAMS WITH TENSION BARS LAPPED IN A REGION OF UNIFORM BENDING MOMENT

1. PRELIMINARY

This part of the investigation was made on 4 rectangular beams under two-point loading. Each beam had a one-half-inch reinforcing bar spliced in the region of uniform bending moment. The length of the lap was in all cases 41 inches (82 diameters) to insure a safe splice. The object in making tests with a lap of this amount was to secure data as a basis for recommendation as to the length of lap necessary (1) when continuous and spliced bars are adjacent, and (2) when all bars are spliced at the same section. There are a number of other conditions which should be studied in this kind of investigation, but it was not feasible to make these studies in connection with this work.

2. DESCRIPTION OF SPECIMENS

Four beams, 10 feet 8 inches long, 10 inches wide, and 12 inches deep, were made. In 3 of the beams the tension reinforcement consisted of 6 plain round bars, extending the full length of the beam (here termed through bars), and 2 short bars (here termed lapped bars), extending from opposite ends of the beam to a point 20½ inches beyond the center of the span. This gave the short bars a lap of 41 inches, which was symmetrical about the center line of the span. In the fourth beam no through

[7] Abrams, Bul. 71, Eng. Exp. Sta., Univ. of Illinois.

bars were present, but the lapped bars were placed in the same relative positions as in the other 3 beams. All lapped bars were one-half inch plain round. All bars were anchored at the ends of the beams by means of semicircular hooks. The lapped bars had no mechanical anchorage at the ends of the laps. The numbers and sizes of bars and the percentage of reinforcement in the several beams are summarized in Table 12.

TABLE 12.—Reinforcement of Beams with Lapped Bars

Beam No.	Through bars		Lapped bars		Reinforcement	Ratio [a] area of through bars to lapped bars
	Number	Diameter	Number	Diameter		
		Inch		Inch	Per cent	
8AAA1	0		2	½	0.2	0
8AAB1	6	½	2	½	1.4	∎
8AAC1	6	⅝	2	½	2.1	10.3
8AAD1	6	¾	2	½	2.9	13.5

[a] Only one lapped bar considered effective for determining this ratio.

For convenience in designating gage lines, the bars were numbered consecutively from 1 to 8, beginning with the bar next to

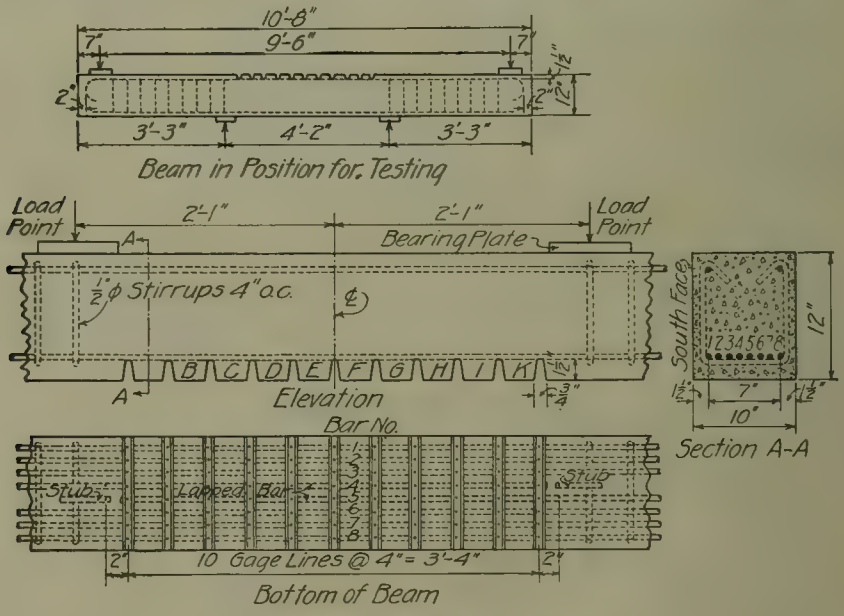


FIG. 39.—*Details of test beams with lapped bars*

the south side of the beam as placed in the testing machine, except for the beam having only the lapped bars. In this case the bars were numbered 4 and 5, the numbers given corresponding

to the numbers of the lapped bars in all the other beams. Fig. 39 shows the arrangement of the reinforcing bars in each beam. To prevent failure by diagonal tension, nine stirrups, made from one-half-inch plain round bars, were placed near both ends of each beam. Exposure at spots on the tension bars, for the purpose of taking strain gage readings, was provided for by placing wooden blocks in the bottom of the forms across the beams at each row of gage points before pouring. These blocks were about three-fourths inch wide and 1½ inches high and extended the full width of the beam. They were tapered slightly to facilitate removal.

The reinforcing steel used was of the quality known as shell discard. Average results of physical tests are given in Table 13.

TABLE 13.—Physical Properties of Reinforcing Steel Used in the Beam Tests

Diameter	Yield point	Ultimate tensile strength	Elongation in 8 inches	Reduction of [illegible]
	Lbs./in.²	Lbs./in.²	Per cent	Per cent
One-half inch	64 000	105 000	16	27
Five-eighths inch	50 000	90 000	[illegible]	46
Three-fourths inch	54 000	93 000	20	21

3. MATERIALS

The concrete was mixed in the approximate proportions of 1:1:1, by volume. The quantities of the materials were determined by weight. A rich mix was chosen in order to make the conditions closely similar in this respect to those in concrete ship construction. The fine aggregate was of two sizes: (1) A bank sand, all of which passed a one-eighth-inch screen; and (2) screenings from the gravel varying from one-eighth to one-fourth inch. The coarse aggregate consisted of pebbles from one-fourth to one-half inch in size. Lehigh Portland cement was used, samples of which were tested at the Bureau of Standards, Washington, D. C., and found to conform to the United States Government specifications for Portland cement.[8]

4. MAKING OF SPECIMENS

The blocks of wood used to expose the steel for strain gage readings were tacked to the bottom of the form, and the tension bars were fastened to them. A steel stub, one-half inch in diameter and 4½ inches long, was placed at the unanchored end of each

[8] Circular of the Bureau of Standards, No. 33.

lapped bar with the intention of taking readings with the strain gage between the stub and the end of the lapped bar. The stirrups were securely wired to the tension reinforcement, and were held in place at the top of the beam by wiring them to a one-half inch plain round bar which extended the full length of the beam. The same form was used for all four beams.

The concrete was mixed in a "Wonder" batch mixer of about 3 cubic feet capacity. Sufficient aggregate for each beam was taken from the storage bin, thoroughly mixed, and its moisture content was determined. Enough water was added to bring the total water content to 13 per cent of the total dry weight of the batch. Each batch was mixed for five minutes at the rate of 12 revolutions per minute, dumped into a tight box, and carried by a traveling crane to the form. As the concrete was shoveled into the form, it was puddled, and the form was jarred with hand hammers. Six 6 by 12 inch cylinders were made with each beam, 3 to be tested at the age of 7 days, and 3 at the same age as the beam when tested. Results of the tests of these specimens are shown in Table 14.

TABLE 14.—Strength of Beams and Control Cylinders

Beam No.	Maximum load [a]	Cylinder strength	
		7 days	27 days
	Pounds	Lbs./in.²	Lbs./in.²
8AAA1	5 600	3195	[illegible]
8AAB1	55 150	[illegible]	[illegible]
8AAC1	51 300	3050	5215
8AAD1	76 400	[illegible]	4575

[a] Beams 28 days old when tested.

The beams were removed from the forms 24 hours after pouring and were covered with wet burlap, which was sprinkled each day until the beams were prepared for the test.

5. METHOD OF TESTING

The tests were made in a 300 000-pound Olsen testing machine. Before placing the specimen in the testing machine, holes were drilled in the reinforcing bars for strain gage readings. The sides and bottom of the beam were whitewashed to facilitate the detection of cracks.

At each gage hole on the lapped bars a spot of whitewash was placed on the exposed bar and on the concrete immediately adjoining it to aid in the detection of slip at the lap during the

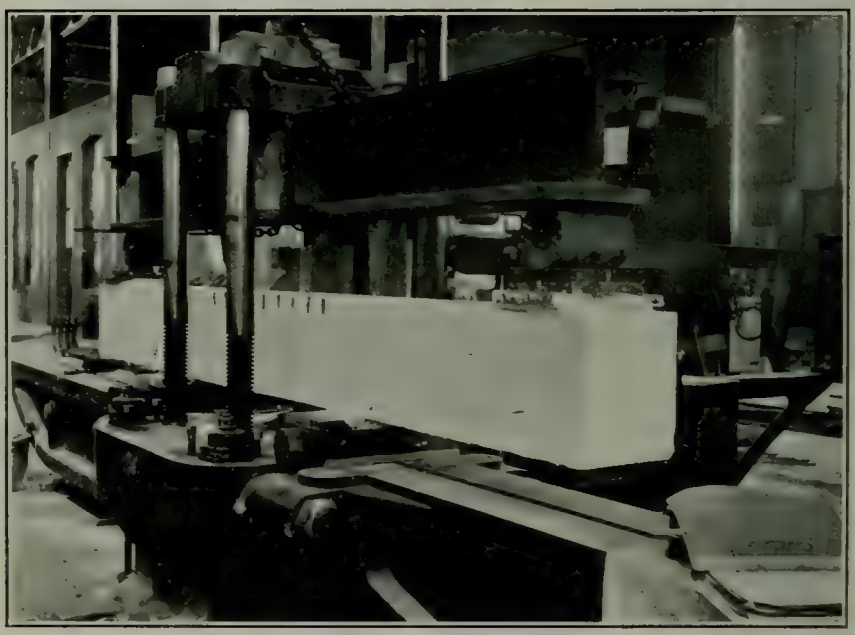


FIG. 40.—*Beam in testing machine ready for application of load*

test. The same procedure was followed on the through bars at the gage holes nearest the load points. It was found that these whitewashed spots gave a sensitive means of detecting any slip, as a minute movement was unfailingly shown by a crack in the whitewash on the sides of the spot parallel to the direction of slipping.

The beams were placed in the testing machine with the tension side uppermost, and were supported on the base of the machine at two points, each of which was 2 feet 1 inch from the center of the beam. They were loaded at the ends of the span through the medium of an H beam attached to the moving head of the testing machine. Fig. 40 is a view of one of the beams in the testing machine.

Since the points of support in these tests corresponded to the load points of beams tested in the normal position, they will be referred to in this paper as load points. The span was 9 feet 6 inches. Deflections were measured at the center of the beam. Readings of deformation were taken with a Berry strain gage on each of the bars in a series of 10 consecutive gage lines, each 4 inches long, 5 gage lines being taken on each side of the center line of the span. For each gage line on a lapped bar there was a gage line beside it on each through bar.

Sections through a beam bisecting a gage line on each bar were designated by letters from A to K, inclusive, except that letter J was omitted. Any gage line is identified by its section letter and bar number; thus K6 indicates the gage line which is cut by section K of bar No. 6. Bar No. 5 ended near section A, and bar No. 4 near section K, beyond the lettered section in each case.

Three sets of strain gage readings were taken at low loads on each beam to furnish a double check on the accuracy of the zero readings. As the test proceeded, strain gage readings were taken at various increments of load; length and direction of cracks were observed with corresponding loads; and the whitewashed observation points were watched to detect any slip of the lapped bars. As the strain gage readings were taken on both sections of the spliced bar at the lap, they are treated as two bars when discussing the phenomena of the lap, but considered as only one bar effective as tension reinforcement.

6. PHENOMENA OF THE TESTS

(*a*) Beam 8AAA1, poured on November 22, 1918, was set up for testing on December 20. This beam had as tension reinforcement two short one-half-inch plain round bars, lapped 41 inches

at the center of the beam, and no through bars. These bars were given the numbers 4 and 5 since they corresponded in positions with bars 4 and 5 of the other beams. Strain gage readings were taken at applied loads of 1000, 2000, 3000, 4000, 5000, and 5600 pounds.

Further details: No cracks appeared until a load of 3000 pounds had been applied. At this load a small tension crack 3½ inches long opened up in the northwest quarter, 20¼ inches from the center of the beam. A similar crack in the same relative position showed in the northeast quarter of the beam. A small crack about 1 inch long showed at the center of the beam. Cracking on the south face of the beam was similar to that on the north face. These cracks started from the depressions left in the concrete by the wooden blocks used to expose the reinforcing bars at the gage holes.

At a load of 4000 pounds the crack in the northwest quarter increased slightly in length. On the southeast face several very fine cracks opened up, showing no connection with each other and no tendency to take the same general direction. Each was about 1½ inches long.

At a load of 5000 pounds the crack in the northwest quarter had extended about 10¼ inches into the beam and had increased very much in width. It showed in similar manner in the southwest quarter. A small crack about 4¼ inches long opened up on the northeast face. After about one hour the load, which had fallen off slightly, was brought up to 5000 pounds, and another set of strain gage readings was taken. No new cracks had appeared. Upon increasing the load to 5300 pounds the tension crack at the east load point opened suddenly, and the load fell off slightly with the machine running; but it immediately picked up and increased to 5600 pounds, which was the maximum load attained. The machine was kept running for some time after the maximum load was reached, the beam holding up a load of 3900 pounds.

After removing the beam from the testing machine, an examination showed that bar No. 4 had slipped 0.26 inch at the unanchored end. On this bar all the whitewashed points showed the bond broken between steel and concrete and also indicated considerable movement of the bar. No. 4 showed movement at all of the observation points except the two nearest the unanchored end. These points were three-fourths inch and 4¾ inches, respectively, from the unanchored end. All of the other whitewashed observation points showed a disturbance of the bond by fine cracks in the whitewash parallel to the bar, but whatever movement there was in the bar was too small to measure by any means available.

(*b*) Beam 8AAB1, poured on December 3, was tested on December 31. The tension reinforcement of this beam consisted of six one-half-inch plain round through bars and two one-half-inch plain round short bars lapped 41 inches at the center of the beam. Strain gage readings were taken at loads of 1000, 3000, 5000, 8000, 22 000, 36 000, 50 000, and 55 150 pounds.

Further details: At a 5000-pound load two very fine cracks showed in the southwest quarter and one in the northwest quarter. These cracks were short tension cracks outside of the load points.

At an 8000-pound load small tension cracks appeared in all the quarters, none of which were over 3 inches long. Very fine cracks appeared at the ends of the lapped bars, also at the center of the beam.

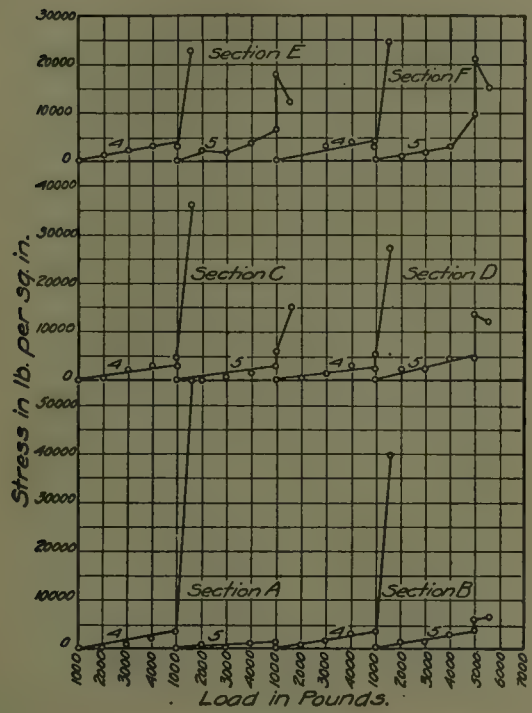


FIG. 41.—*Beam 8AAA1; lapped bars only. Sections A–F*

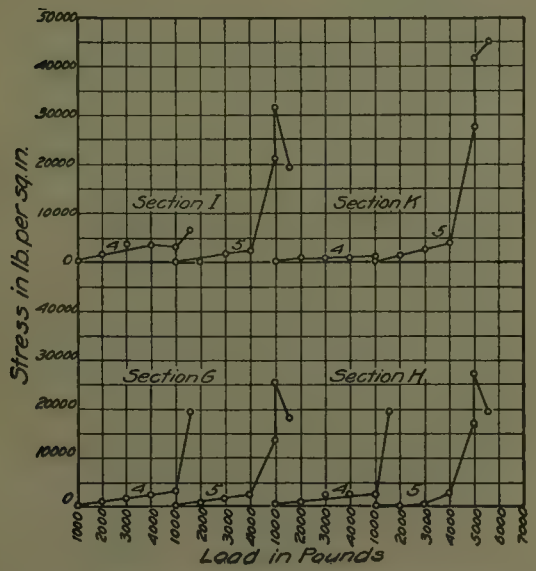


FIG. 42.—*Beam 8AAA1; lapped bars only. Sections G–K*

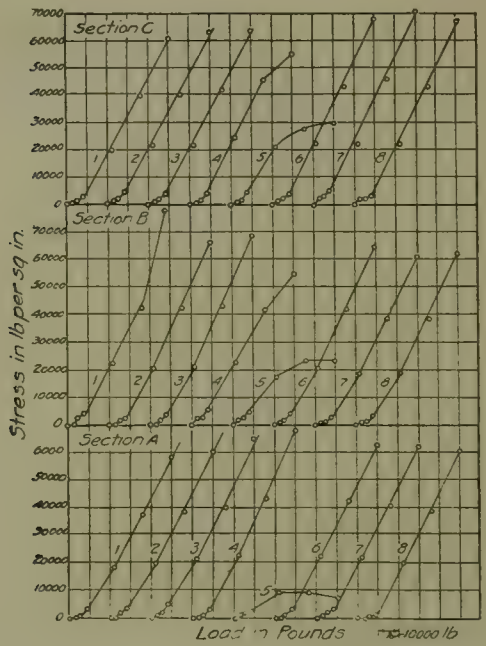


FIG. 43.—*Beam 8AABI; one-half inch through and one-half inch lapped bars. Sections A–C*

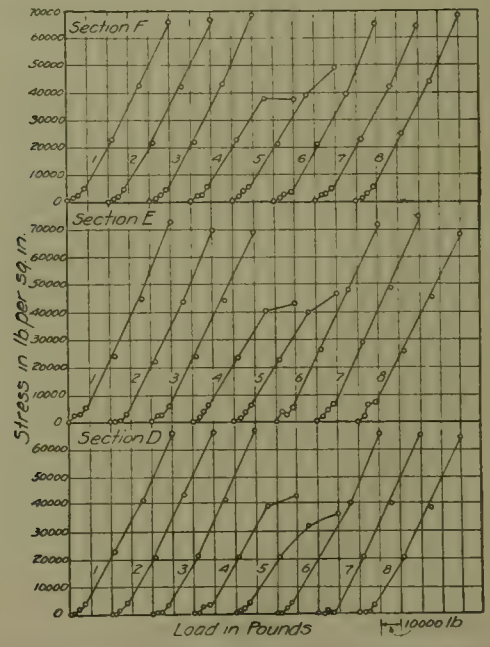


FIG. 44.—*Beam 8AABI; one-half inch through and one-half inch lapped bars. Sections D–F*

At a 22 000-pound load cracks opened up uniformly in all quarters and extended up into the beam about 7 inches. Between the load points tension cracks were quite uniformly spaced and extended into the beams about 6½ inches. No disturbance of the bond of the lapped bars could be seen at any of the observation points.

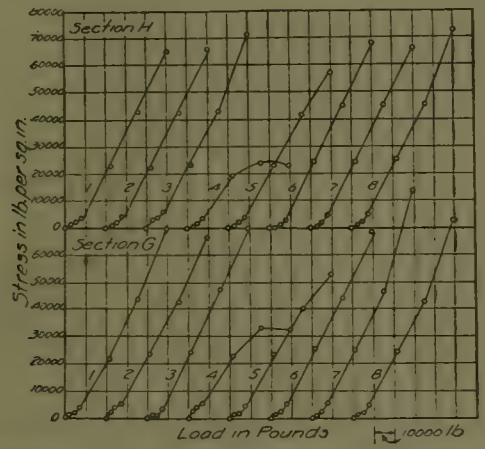


FIG. 45.—*Beam 8AAB1; one-half inch through and one-half inch lapped bars. Sections G–H*

At a load of 36 000 pounds all the cracks had lengthened an appreciable amount, but showed no great increase in width. Bar No. 4 showed the bond broken at the first two observation points on the unanchored end at readings K4 and I4. None of the other points showed any movement.

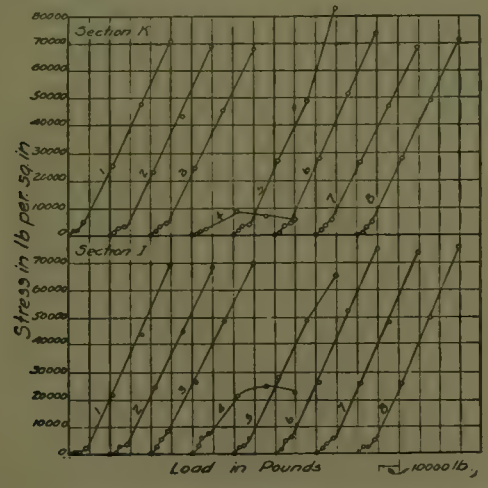


FIG. 46.—*Beam 8AAB1; one-half inch through and one-half inch lapped bars. Sections I–K*

At a 50 000-pound load the observation points on No. 4 lapped bar showed movement from the unanchored end to 4 inches beyond the center of the beam. On No. 5 bar the observation points from the unanchored end to the center of the beam showed movement. All of the cracks had increased in width, particularly the one

at the east end of the lapped bar. The cracks outside of the load points had the direction of diagonal tension cracks.

The load increased to 55 150 pounds and then dropped slightly, and it was evident from the width of the tension cracks that the yield point of the steel had been reached. Strain gage readings were taken. The No. 4 bar showed slip from the unanchored end to 12 inches beyond the center of the beam. Bar No. 5 showed movement at all the observation points to the center of the beam. The load continued at about 54 000 pounds for some time, falling off very slowly with the machine running. The test was discontinued after the center deflection had reached 1.35 inches. The observation points on bar No. 5 from the unanchored end to 12 inches beyond the center of the beam and those on bar No. 4 from the unanchored end to 16 inches beyond the center of the beam showed movement. No slip of the through bars was observed.

(*c*) Beam 8AAC1, poured on November 29, was made ready for test December 27. This beam had six five-eighths-inch plain round through bars and two short sections of one-half-inch plain round bars lapped at the center of the beam for tension reinforcement. Strain gage readings were taken at loads of 1000, 3000, 5000, 10 000, 30 000, 50 000, and 51 000 pounds.

Further details: At a load of 3000 pounds one small crack developed in the southeast quarter at the end of the lapped bar.

At the 5000-pound load a similar crack showed in the northeast quarter.

At the 10 000-pound load a small crack in the southeast quarter extended about 3 inches parallel to the horizontal steel. Small tension cracks showed in all quarters between the load points and the supports.

At the load of 30 000 pounds several cracks appeared between the load points extending about halfway up the sides of the beam. Outside of the load points there were two or three cracks in each quarter, those in the northwest quarter having the direction of diagonal tension cracks. None of the observation points, consisting of patches of whitewash on the bars, showed disturbance of bond. The crack in the southeast quarter parallel to the horizontal bars showed a slight extension, and most of the cracks in all of the quarters showed branches parallel to the horizontal bars.

At a 50 000-pound load diagonal tension cracks showed in all the quarters. The cracks between the load points had opened wider and extended deeper into the beam. Bar No. 5 (lapped) showed slip at all the observation points from the free end to 8 inches beyond the center of the beam. Bar No. 4 showed movement at all the points from the free end to the center of the beam. The outside through bar on the north side showed slip at the observation point nearest the west load point. Horizontal cracks along the outside bars between load points became somewhat longer. Also outside of load points horizontal cracks extended toward the ends of the beams.

On starting the testing machine the load increased to 51 300 pounds, then dropped to 51 000 pounds. The size of the cracks showed that the yield point of the steel had been reached. A set of strain gage readings was taken. The first observation point at the unanchored end of bar No. 4 was the only one unbroken on the lapped bars. The whitewashed observation points were broken on the two outside bars at readings A7 and A8, also at K1, K2, K3, K5, and K8, showing the slip of these bars.

Loading was continued, and cracks opened up uniformly between the load points. The load continued at about 50 000 pounds for some time. After the center deflection had reached 0.87 inch the test was discontinued. The load at this point was 49 700 pounds.

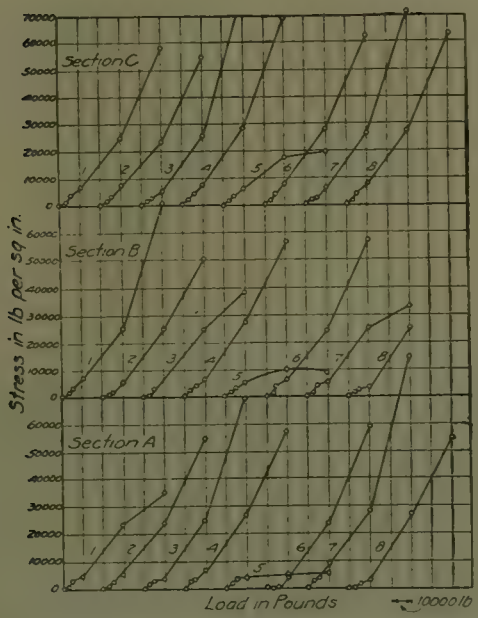


FIG. 47.—*Beam 8AAC1; five-eighths inch through and one-half inch lapped bars. Sections A-C*

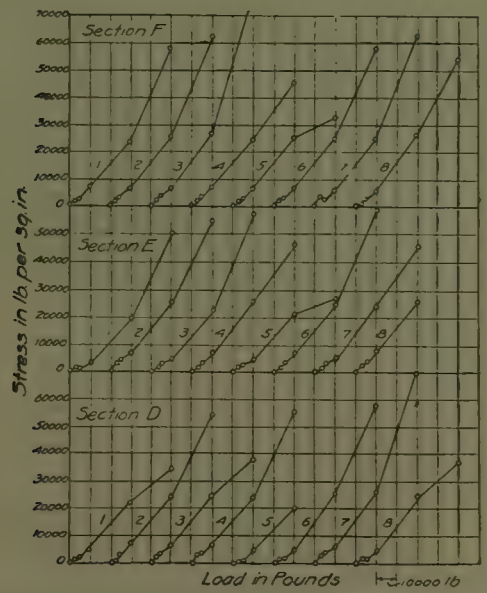


FIG. 48.—*Beam 8AAC1; five-eighths inch through and one-half inch lapped bars. Sections D-F*

(*d*) Beam 8AAD1, poured on November 26, was set up for test December 24. This beam had for tension reinforcement six three-fourths-inch plain round through bars, anchored at both

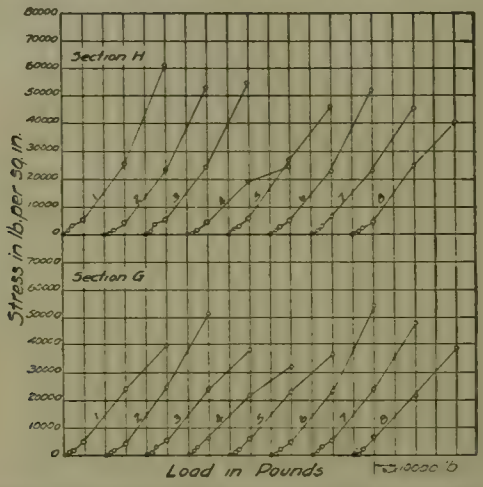


FIG. 49.—*Beam 8AAC1; five-eighths inch through and one-half inch lapped bars. Sections G–H*

ends of the beam, and two short sections of one-half-inch plain round bars lapped at the center of the beam. On removing the wooden blocks to drill the reinforcing bars for strain gage readings,

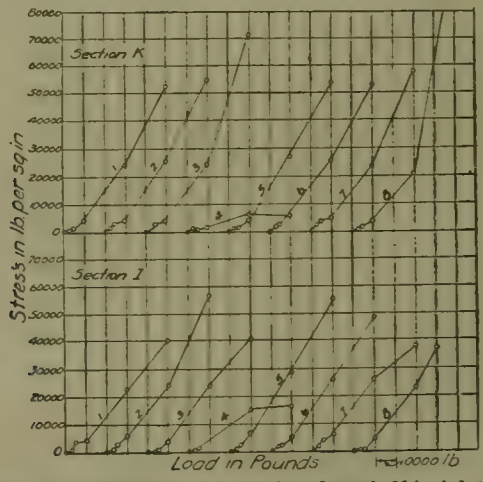


FIG. 50.—*Beam 8AAC1; five-eighths inch through and one-half inch lapped bars. Sections I–K*

the concrete around the bars was found to be porous. Fig. 51 shows a view of the bottom of this beam. All the concrete between the blocks was removed and a rich mortar carefully placed around the bars and rounded off above them. This was done a

FIG. 51.—*Porous condition around reinforcing bars of beam 8AAD1*

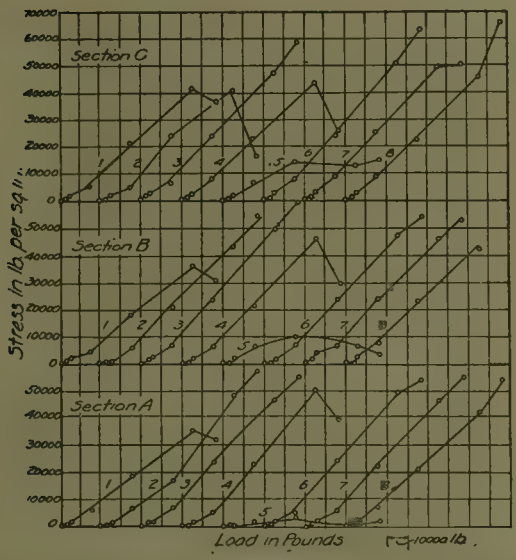


FIG. 52.—*Beam 8AAD1; three-fourths inch through and one-half inch lapped bars. Sections A–C*

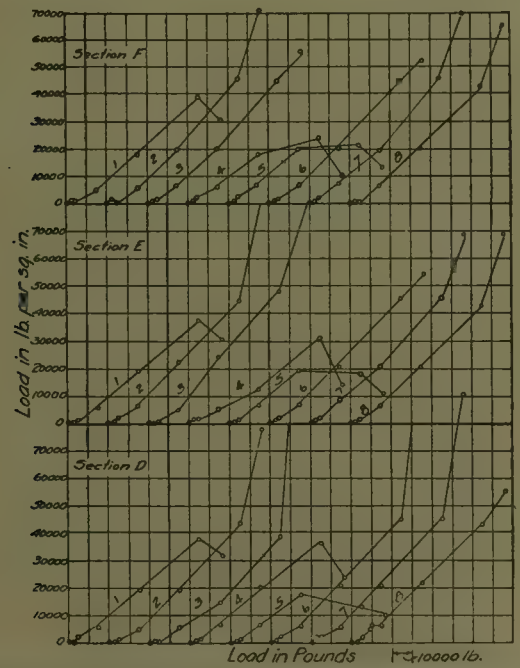


FIG. 53.—*Beam 8AAD1; three-fourths inch through and one-half inch lapped bars. Sections D–F*

week before the test. Strain gage readings were taken at loads of 1000, 3000, 5000, 15 000, 35 000, 65 000, 76 400, and 73 000 pounds.

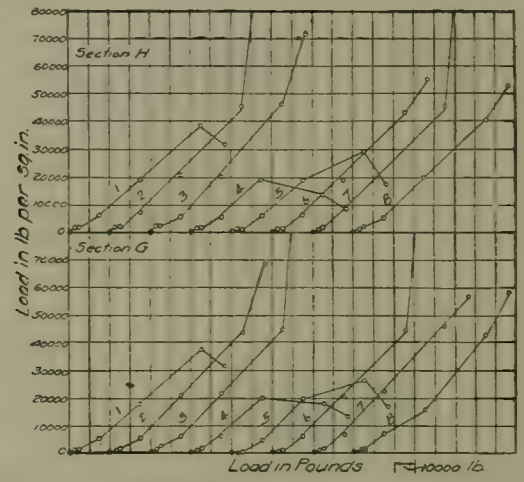


FIG. 54.—*Beam 8AAD1; three-fourths inch through and one-half inch lapped bars. Sections G–H.*

Further details: At a load of 3000 pounds fine cracks showed in the sections at the free ends of the lapped bars.

At a load of 5000 pounds more fine cracks showed, but except for one on the southeast face, none were over 2 inches long.

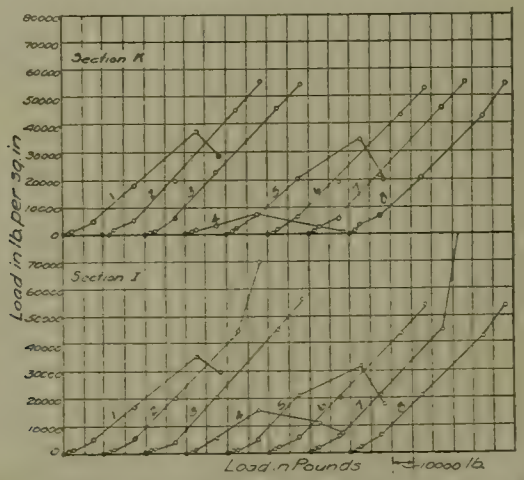


FIG. 55.—*Beam 8AAD1; three-fourths inch through and one-half inch lapped bars. Sections I–K*

At a load of 15 000 pounds cracks showed considerable development. Horizontal cracks appeared along the outside tension bars at each end of the beam. No breaking of the bond at the observation points was observed at this load.

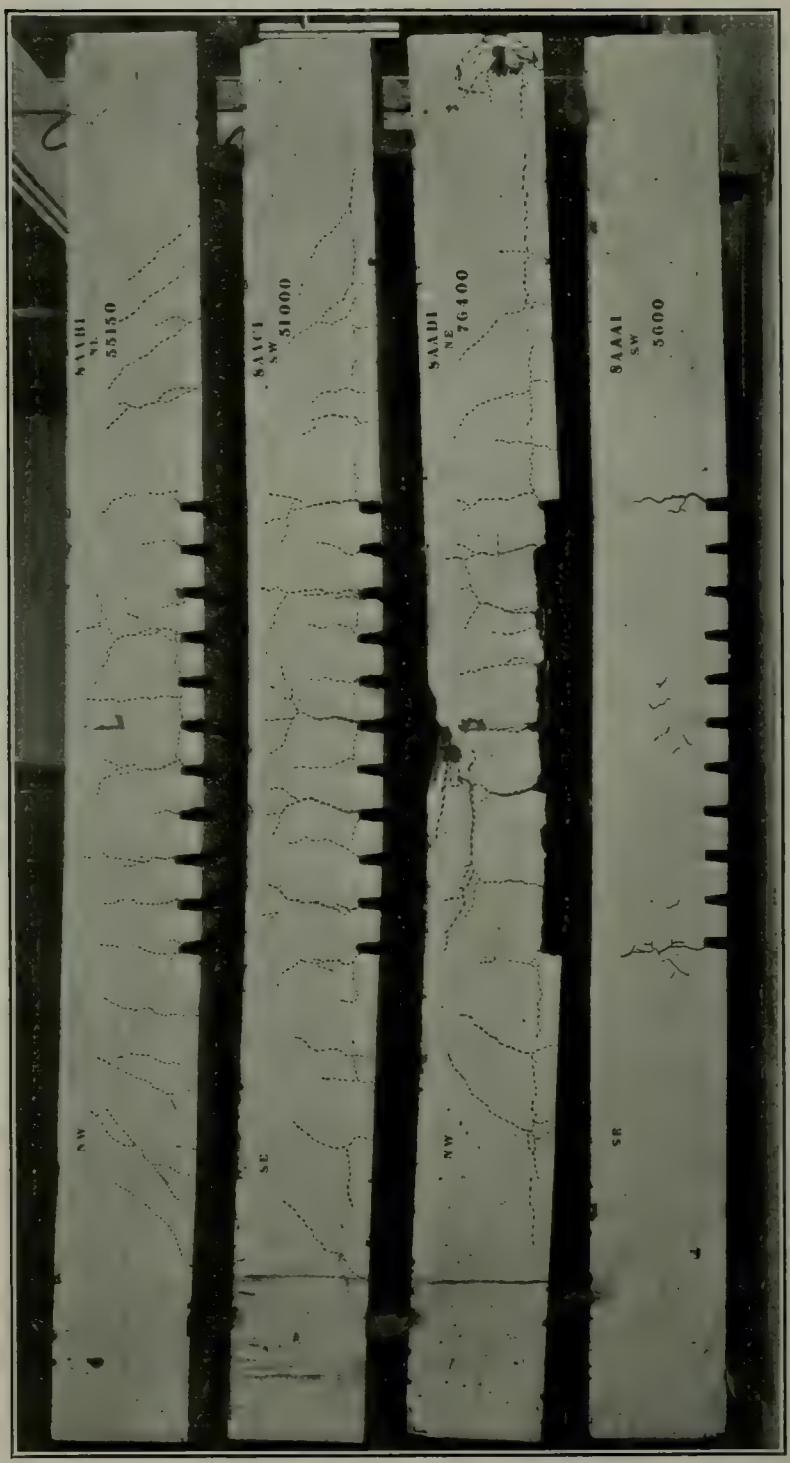


FIG. 56.—*Beams after failure, showing crack development*

At a load of 35 000 pounds diagonal tension cracks showed in all quarters, and tension cracks appeared at the center of the beam. The first two observation points at the free end of each of the lapped bars showed slip.

With a load of 50 000 pounds on the beam, the whitewashed observation points showed movement of the lapped bars for four consecutive points from the free ends of the bars.

At a load of 65 000 pounds slip was observed at seven consecutive points. No movement of the through bars was observed. While standing at the load of 65 000 pounds, two more observation points on bar No. 5 and one more on bar No. 4 showed movement.

At a load of 76 400 pounds cracks following the outer horizontal bars showed throughout the full length of the bars, following around the hooks at the southwest and northeast corners. The whitewashed observation points showed that the two outer through bars had slipped. One of the lapped bars showed slip at all the points of observation. The other bar showed slip at all but the observation point nearest the free end. The concrete started to split off at the southwest corner around the hooked end of the outer through bar. There was originally a three-thirty-seconds-inch covering of concrete over the bar at this point. This reading was taken at the maximum load. Although the shearing stress at this load was 465 pounds per square inch there was no indication that failure due to this cause was approaching.

The load dropped off to 73 000 pounds and remained at this point for some time with the machine running. The size of cracks indicated that the stress in the steel was reaching the yield point, and so a set of readings was taken. The readings at this load showed the steel to be overstressed in the locality of these cracks.

Loading was resumed at constant speed. The load fell off to 68 000 pounds and continued at this point for some time, or until a failure by compression of the concrete on the top of the beam at the center of the span occurred.

This failure was sudden and accompanied by a sharp report. On the northeast corner the concrete had spalled off in about the same manner as at the southwest corner, due to slipping of the hooked end of the outside bar. The other corners were not affected. The other three-fourths-inch bars did not show any failure of bond except at the region where the stress exceeded the yield point of the steel. All the points of observation on the lapped bars which were covered with whitewash showed movement. The movement of the ends of these bars was over one-half inch.

7. GENERAL RESULTS

Table 14 gives some of the results of these tests.

Fig. 56 shows a view of all the beams after test. The cracks are shown by broken lines. Fig. 63 gives the load-deflection diagrams for all the beams. Figs. 43 to 56 give load-stress diagrams for all the gage lines for various sections of the portions of the beams under uniform bending moment.

8. DISTRIBUTION OF TENSILE STRESS AMONG BARS

Figs. 57 to 62 give the intensity of tensile stress among the bars across the beams at various sections. Two loads are represented in each case. The average tensile stress for all bars at each load is shown by a broken line. A study of these diagrams shows that there was a point on each lapped bar beyond which there was a falling off of the stress due to the slipping of the free end of the bar. Proceeding from this point toward the end of the bar, the intensity of stress steadily decreased, and the stress in the other

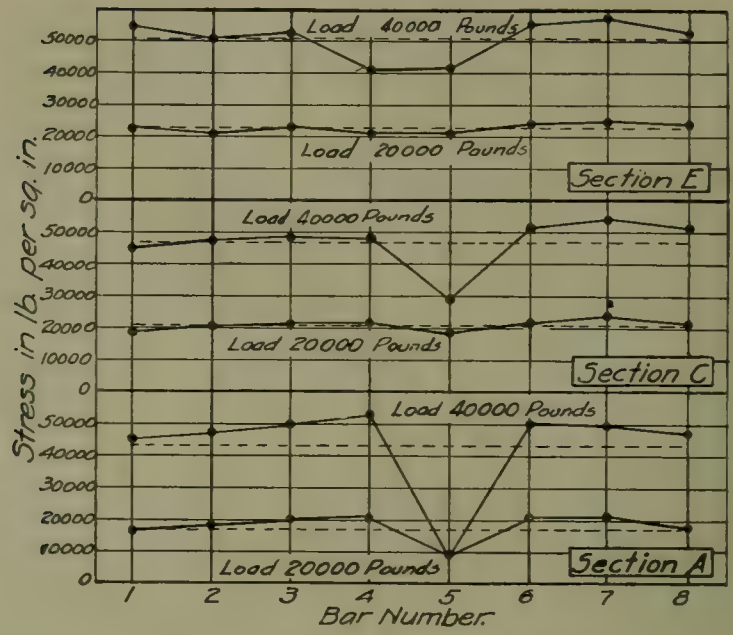


FIG. 57.—*Beam 8AABI. Sections A, C, and E*

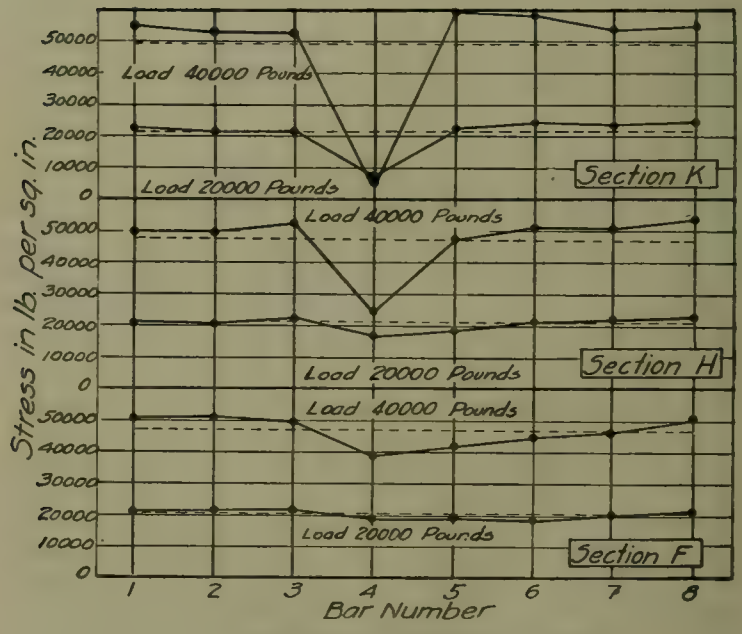


FIG. 58.—*Beam 8AABI. Sections F, H, and K*

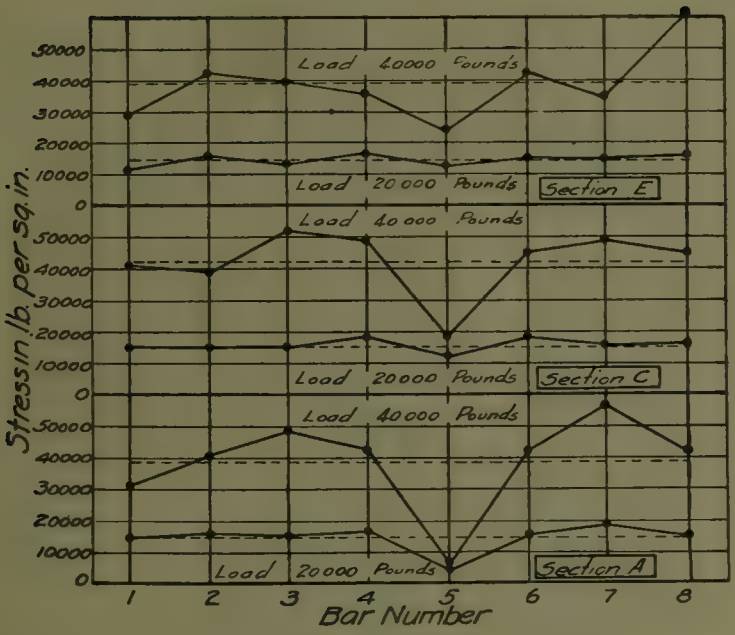


FIG. 59.—*Beam 8AAC1. Sections A, C, and E*

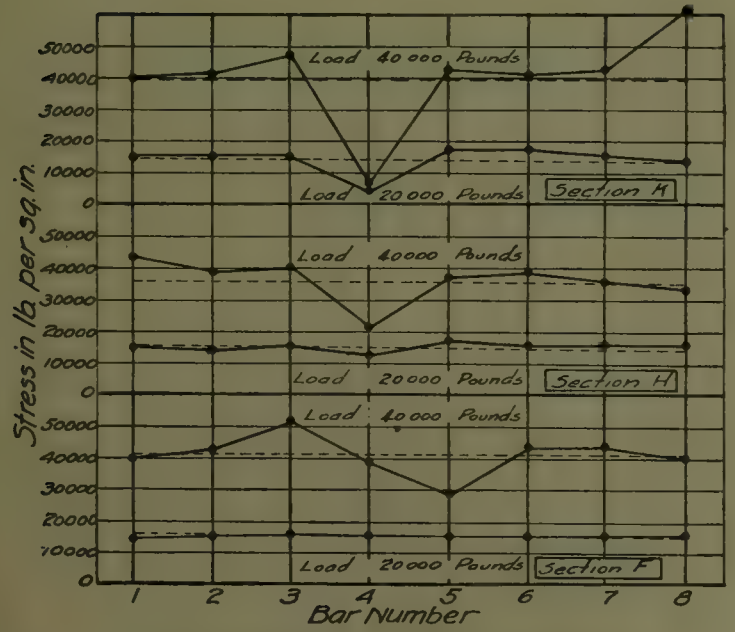


FIG. 60.—*Beam 8AAC1. Sections F, H, and K*

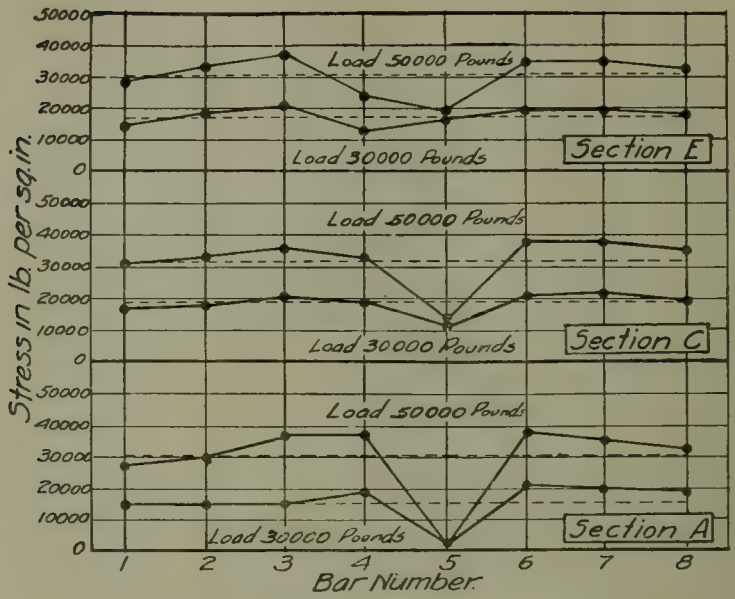


FIG. 61.—*Beam 8AADI. Sections A, C, and E*

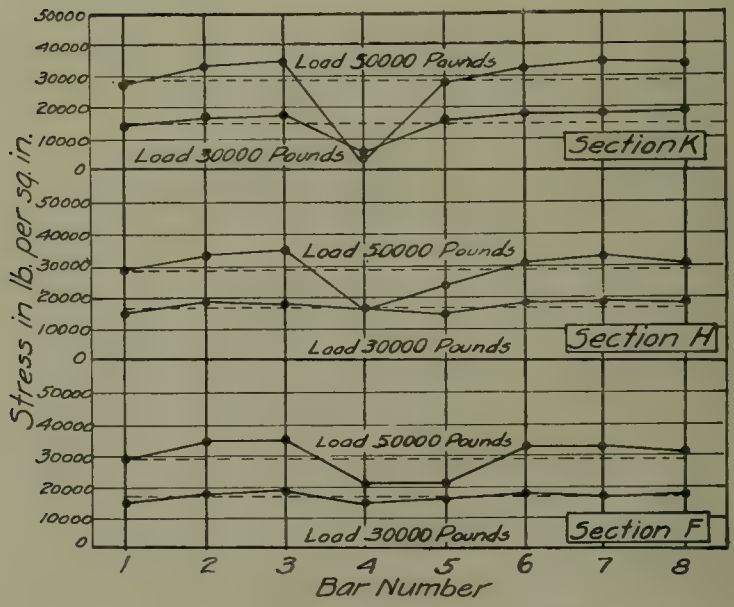


FIG. 62.—*Beam 8AADI. Sections F, H, and K*

bars of the beams increased correspondingly at the same sections. In most cases the increase in stress was greater in the through bars immediately adjacent to the lapped bars than in the other bars of the beam. However, this tendency is not pronounced, and there is no evidence that it is affected by the variation in the proportional areas of through and of lapped bars.

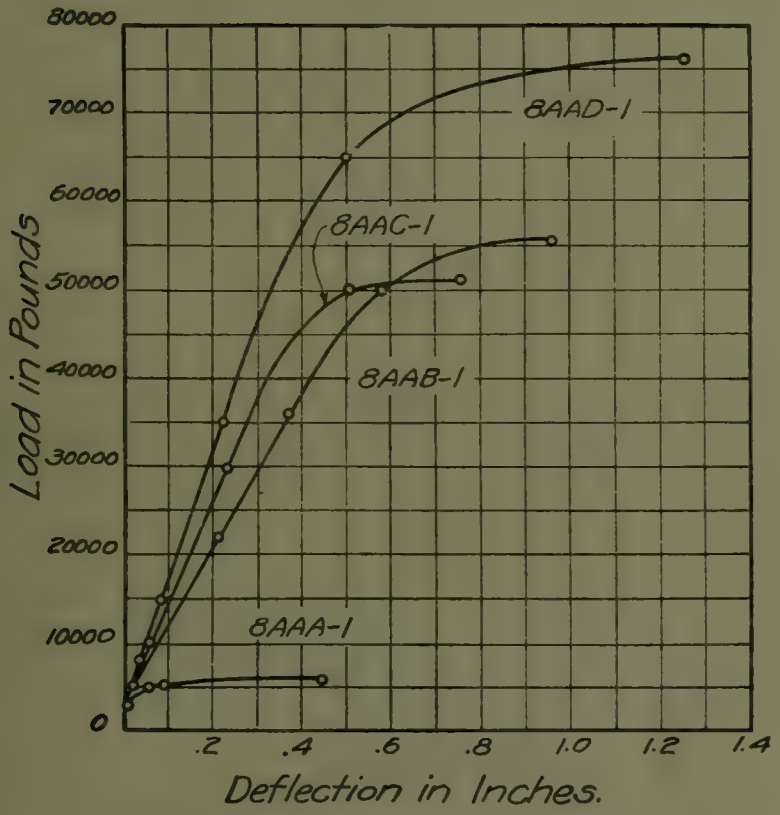


FIG. 63.—*Load-deflection diagrams for all beams*

9. DISTRIBUTION OF TENSILE STRESS ALONG BARS

Figs. 64 and 65 show the distribution of tensile stress as measured on the lapped bars for each of the beams. For the beams with through bars, the average tensile stress for all the through bars at the corresponding sections is shown by a broken line. Curves are given for loads giving computed stresses of 16 000 and 40 000 pounds per square inch in the tension bars.

The curve for beam 8AAD1, Fig. 65, at a computed stress of 40 000 pounds per square inch, shows that the stress in one of the bars of the lap did not become equal to that in the adjacent through bars within the length of the splice. This was probably

due to the porous condition of the original concrete around the tension reinforcement of this beam.

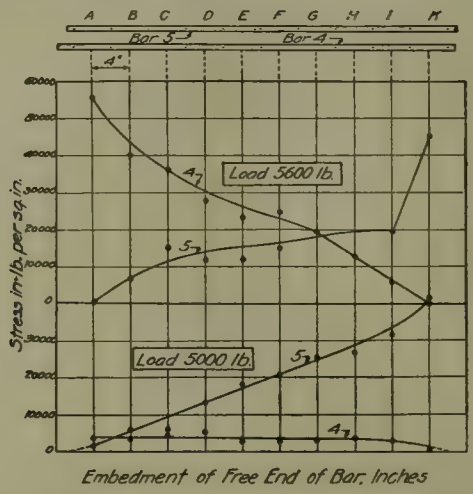


FIG. 64.—*Distribution of tensile stress along lapped bar in beam 8AAA1*

The curves for beam 8AAA1, Fig. 64, are for a higher computed stress than 40 000 pounds per square inch. At the load which gives 40 000 pounds per square inch computed stress, cracking had

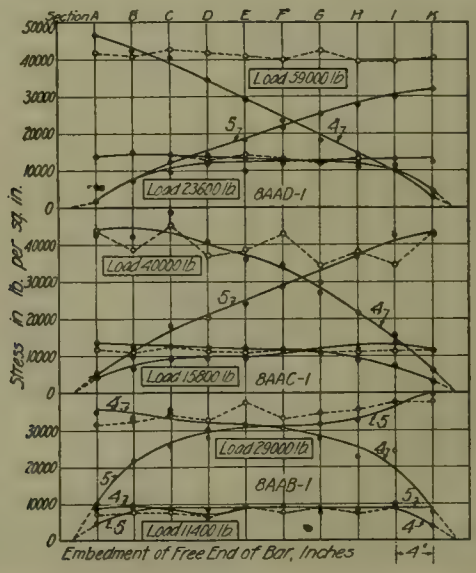


FIG. 65.—*Distribution of tensile stress along lapped bar in beams 8AAB1, 8AAC1, and 8AAD1*

not developed sufficiently to cause the tension reinforcement to carry all the tensile stress unaided by the concrete.

10. DISTRIBUTION OF BOND STRESS ALONG BARS

Figs. 66 and 67 show the distribution of bond stress along the bars of the lap for the three beams with through bars. Each curve is the average for the two lapped bars of a beam. The values plotted are proportional to the differences in tensile stress as measured by the strain gage on consecutive gage lines, starting at the free end of the bar. Curves for two loads are given. The curve for the lower load of each beam shows the distribution of bond stress along the bar when the bond stress at the free end of the bar is at or near the maximum. The curves for the higher loads

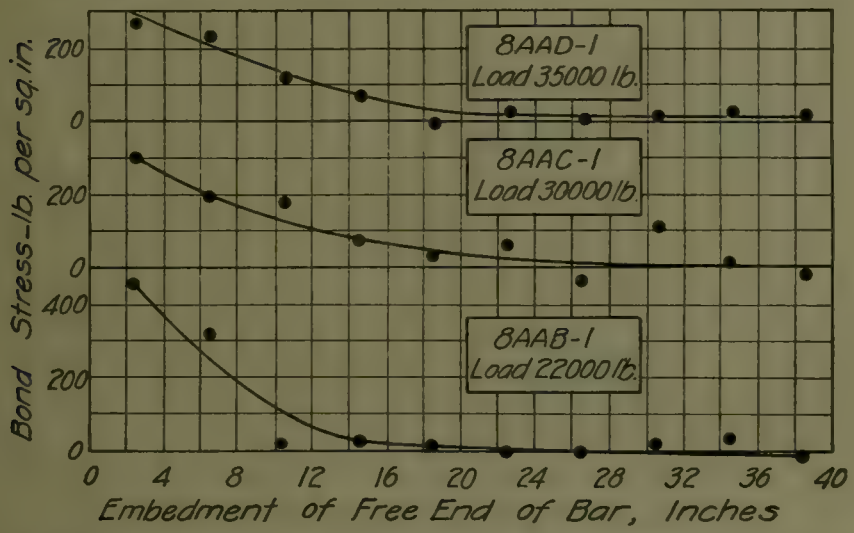


FIG. 66.—*Distribution of bond stress along lapped bars in beams 8AAB1, 8AAC1, and 8AAD1*

have the same general form, but here the bond stress does not decrease to zero before the end of the lap of the adjacent bar is reached.

In the recorded bond stresses no correction has been made for the area of steel left exposed by the wooden blocks placed in the concrete for the purpose of locating the strain gage readings on the steel. This area would amount to about 5 per cent of the total surface area of the steel between gage points. Near the unanchored end of the lapped bars individual maximum bond stress values may be found which are about 75 per cent of the average values given in the first part of this report for pull-out tests of uncoated plain square bars.

11. SLIP OF BARS

Fig. 68 shows the progress of slip along the bars of the lap from the free ends to the ends of the splice for the same loads as those for which the bond-stress curves are given. Each curve is the average for the two lapped bars of a beam.

The slip for any point on the lapped bar at any load was obtained by subtracting the sum of the measured deformations between that point and a point where evidently no slip had oc-

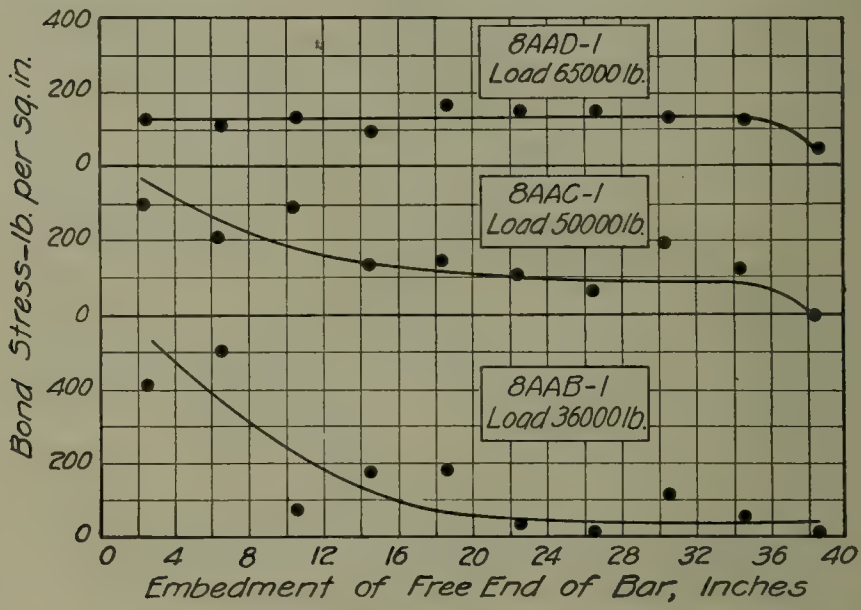


FIG. 67.—*Distribution of bond stress along lapped bars in beams 8AAB1, 8AAC1, and 8AAD1 at higher loads*

curred from the sum of the corresponding deformations of the adjacent through bars. The original plan of measuring the slip of the end of the lapped bar was not used on account of the fact that in all cases a crack opened between the stub and the end of the bar.. This caused an indeterminate error in the measurement of the slip, as taken by the strain gage, equal to the width of the crack.

A comparison of the bond-stress curves for the load which gives the maximum bond stress at the end of the lapped bar, Fig. 66, with the slip curves for the same loads, Fig. 68, shows that the maximum bond stress at the end of the bar occurred at a very small slip. However, the bond stress at the end of the bar fell off slowly from the maximum, and with a slip twice as great

as that which gave the maximum the bond stress was not very much reduced. This action is evidently due to frictional bond resistance. The slipping of the hooked bars of beam 8AAD1

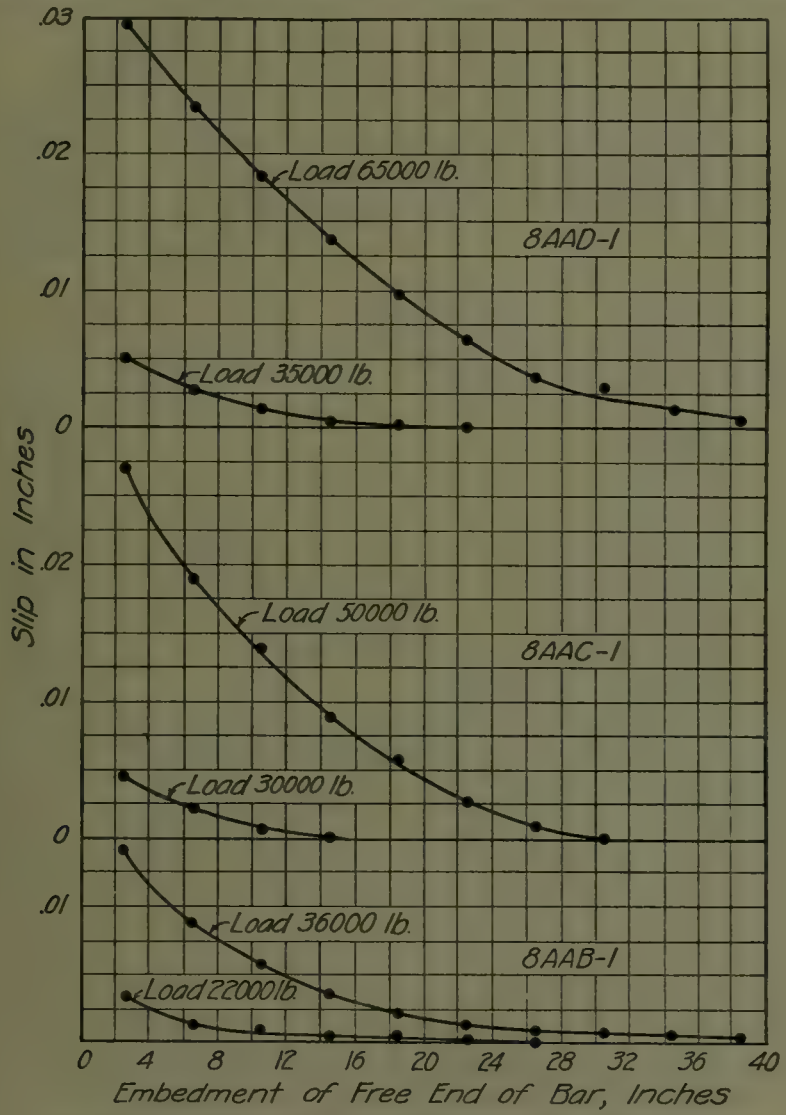


FIG. 68.—*Distribution of slip along lapped bars in beams 8AAB1, 8AAC1, and 8AAD1*

at the maximum load indicates that the bond resistance was insufficient to develop the full tension which existed in the bar and that the hooks were coming into action in furnishing anchorage. The computed bond stress at the maximum load was about

300 pounds per square inch or 6 per cent of the compressive strength of the concrete and the computed tensile stress was about 62 000 pounds per square inch. The lack of concrete in contact with the bars over a considerable distance probably contributed to this slipping. The condition of this beam is shown in Fig. 51. The same figure indicates that at least in the middle portion of the beam the outside bars were much closer to the sides of the beam than is called for in Fig. 39. If the condition at the end of the beam was similar to that at the center of the span, the bars were closer to the side of the beam than ordinary good practice would approve and this defect is probably responsible for the bursting of the hooked end of the bar through the side of the beam.

12. LENGTH OF LAP REQUIRED FOR BEAMS WITH THROUGH BARS

To serve as a basis for analysis, it may be assumed that for any given tensile stress the safe length of lap is the distance from the free end of the bar to the point where the tensile stress in the lapped bar becomes constant or equal to the stress in an adjacent through bar. If the lap furnished is less than this, the tensile stresses in the through bars at the ends of the lap must be greater than the value computed by the equation

$$f_s = M/A_s jd,$$

unless the difference in stress is taken up by the concrete in tension. This is based on the assumption that the entire lap lies within a region of constant bending moment.

With this assumption as a basis, Fig. 69 has been prepared. The data from the beam without through bars were not used in this figure. The amount of reinforcement for this beam was very small; consequently, the concrete took a large portion of the tensile stress, and the stress curves for the steel were too erratic for use in this way. The ordinates of the points given represent the distance from the ends of the lapped bars at which the measured stresses shown as abscissas were developed. A straight line represents fairly well the mean of the values shown by the points. The equation of this line is

$s = 0.00059\ f_s$, where (1)

s = distance in inches from end of lapped bar to a point where the bond stress becomes zero under a given load, and

f_s = the tensile unit stress computed from the corresponding bending moment.

It is possible that further investigation, using plain bars of diameters other than one-half inch, or deformed bars, would yield a different constant in this equation. A further modification would probably be produced by using concrete mixed in other proportions than 1 : 1 : 1.

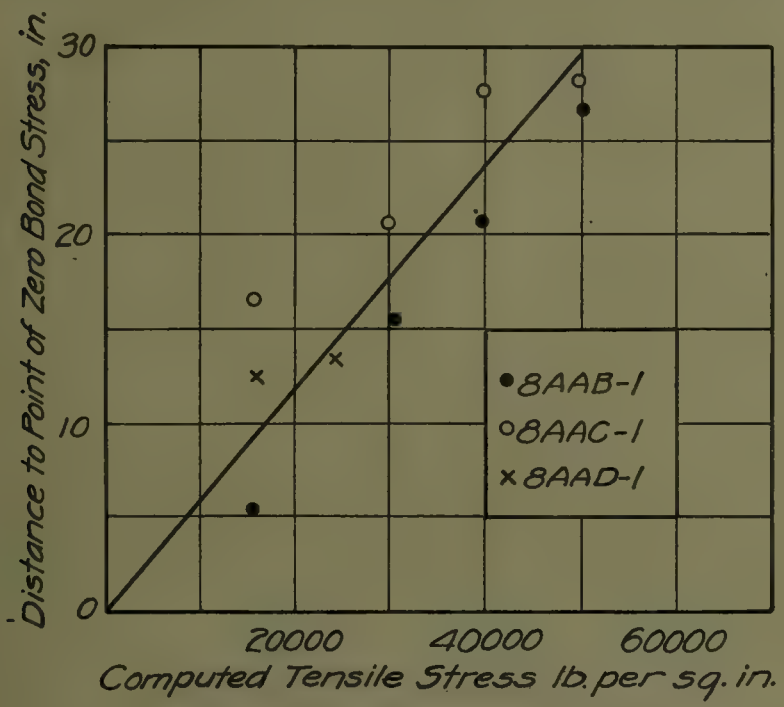


FIG. 69.—*Relation of tensile stress to length of lap required*

It is apparent that the total tensile stress in a lapped bar at the section where the bond stress becomes zero is

$$\frac{\pi a^2 f_s}{4} = \pi a u s, \text{ where} \qquad (2)$$

a = diameter of bar, and
u = average bond stress in distance s.

From this equation,

$$f_s = \frac{4\ us}{a}. \qquad (3)$$

Substituting in equation (1)

$$s = 0.00059\ \frac{4\ us}{a}, \text{ and} \qquad (4)$$

$$u = \frac{a}{4 \times 0.00059}. \qquad (5)$$

For lapped bars one-half inch in diameter the value of u from this equation equals 210 pounds per square inch. Equation (5) indicates that the value of the bond stress was independent of the tensile stress developed. The acceptance of this form of equation is equivalent to concluding that the average bond stress over the length s was the same at all stages of the test, but that the length s increased as the tensile stress increased.

Reference to beam 8AAB1, Figs. 66 and 67, shows that the distance s from the end of the bar at which the bond stress approximates zero was greater for the load of 36 000 pounds than for the load of 22 000 pounds, but that the average bond stress was not far from 210 pounds per square inch in both cases. For beams 8AAC1 and 8AAD1 the average bond stress was less than this. Apparently the conclusion that the average bond stress developed over the length of lap is independent of the tensile stress developed in the bars is not fully justified, but as a basis for design this assumption is probably exact enough.

It is generally assumed that the length of embedment required to develop the tensile strength of a bar is directly proportional to the diameter of the bar. In this investigation it was not feasible to test a sufficient number of beams with lapped bars to confirm this assumption, but it is here accepted as logically to be expected. Since these tests were made on one-half-inch bars, the equation becomes by substitution from equation (1),

$$\frac{s}{a}=\frac{0.00059}{\frac{1}{2}}f_s=0.00118\,f_s, \tag{6}$$

or,

$$s=0.00118\,f_s a. \tag{7}$$

The stress in the lapped bar just outside the lap is the f_s of this equation. If this is less than the average stress at this section calculated by the equation

$$f_s=\frac{M}{A_s\,jd},$$

then the stress in the through bars must be greater unless the difference is taken up by the tensile resistance of the concrete. Consequently, in using equation (7) to compute the minimum safe length of lap, the value of f_s should be the yield point of the steel. If f_s be taken less than this, the yield point will be passed in the through bars before it is reached in the lapped bars. Assuming the yield point to be 40 000 pounds per square inch, and that the concrete has the same strength as the concrete used in these tests,

$$s=47.2a. \tag{8}$$

Under a working load the total tensile stress developed in the lapped bar within the distance s from its free end is as stated in equation (2). In this case the values of u and s to be used are the working stress. From this equation, using $f_s = 16\,000$,

$$u = \frac{af_s}{4s} = 212 \text{ pounds per square inch.}$$

From Table 14 the average compressive strength of the control cylinders was 5020 pounds per square inch. Assuming that the bond resistance is proportional to the compressive strength of the concrete,[9] f'_c,

$$\frac{u}{f'_c} = \frac{85}{5020} = 0.0169, \text{ and}$$

$$u = 0.0169\, f'_c \qquad (9)$$

13. EFFECT OF VARIATION OF AREA OF THROUGH BARS ON LENGTH OF LAP REQUIRED

A study of Fig. 69 indicates that within the limits of the data used for this diagram a variation in the ratio of the area of the through bars to that of the lapped bars had little if any effect on the length of lap required. However, in these tests the least area of through bars was six times that of the lapped bars. With smaller proportions of through bars it is possible that a greater length of lap might have been needed. An extreme case is represented by beam 8AAA1, which had no through bars. This beam failed by the pulling out of one of the bars, although the lap was 80 diameters. This occurred at a load which gave a computed tensile stress in the steel of 50 000 pounds per square inch, and a measured stress of 55 000 pounds per square inch at one point. While this stress was high, it was less than the yield point of the steel. The evidence afforded by the failure of beam 8AAA1 indicates that a greater length of lap is required for beams in which all the bars are lapped than for beams in which there are through bars. Further investigation along this line is desirable.

14. SUMMARY OF RESULTS

The following statements summarize the results of the beam tests reported above:

(*a*) Proceeding along a lapped bar toward its unanchored end from the point where the stress in it begins to diminish, the stress lost is picked up by the other bars of the beam. In a majority of

[9] Abrams, Tests of Bond Between Steel and Concrete, Bull. 71, Eng. Exp. Sta., Univ. of Illinois, p. 94, 1913.

cases a larger amount of the stress lost by the lapped bar was picked up by bars immediately adjacent than by the other bars of the beam.

(*b*) The maximum bond stress developed by the lapped bars was about 75 per cent of that reported for pull-out tests of uncoated plain square bars in Division II of this paper. The maximum bond stress occurred at a smaller amount of slip of bar for the beams than for the pull-out tests.

(*c*) The average bond stress in the lapped bars was practically independent of the tensile stress in the bars, but the length over which it was effective varied with the magnitude of the stress.

(*d*) For the purpose of design the minimum safe length of lap may be taken as the distance from the unanchored end of the lapped bar to a point on the bar where the bond stress is zero, when the tensile stress in the steel is at the yield point. With this assumption the tests indicate that using steel which has a yield point stress of 40 000 pounds per square inch, and with continuous bars adjacent to the lapped bars and with concrete of the grade here used, the lap should be about 48 diameters. Care should be used in applying this relation under other conditions.

(*e*) The tests of beams with through bars do not show that differences in the ratio of the area of the through bars to the total area cause any variation in the length of lap required. The test of the beam with no through bars indicated that when all the bars are lapped, a longer lap is needed than when through bars are present. Further investigation is needed on this phase of the subject.

(*f*) A shearing stress of 465 pounds per square inch was developed in one of the beams without sign of approaching failure by diagonal tension.

IV. TESTS ON STIRRUP PULL-OUT SPECIMENS

1. PRELIMINARY

The tests described in the following pages of this paper were made on 12 test specimens designed to furnish useful information on the strength of stirrups. The dimensions of the specimens and reinforcement used conformed to a typical section through the shell and frame in a concrete oil-tank ship which was built by the Emergency Fleet Corporation. In such a section, due to greater stiffness in the frame than in the shell, the hydrostatic pressure of the cargo tends to force the shell away from the frame, thus

adding direct tension to the tension induced in the stirrups by shearing stresses in the frame. Under such circumstances the efficiency of the anchorage of the stirrups is of great importance.

2. DESCRIPTION OF SPECIMENS

The specimen consisted of a concrete slab 4 inches thick, cast monolithic, with a concrete rib 6½ inches wide and 12 inches deep. The slab was 1 foot 10½ inches long and 1 foot 4 inches wide. The rib was 2 feet 4 inches long and was attached to the slab along its center line in the short direction; it projected 6

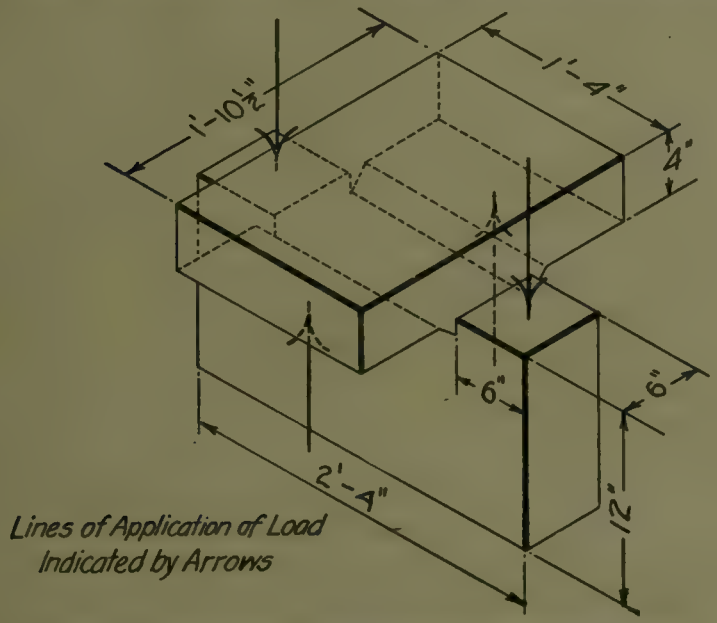


FIG. 70.—*Isometric view of specimen used in stirrup pull-out tests*

inches beyond the slab at each end to afford a bearing for the downward load. Fig. 70 is an isometric view of the test specimen.

The reinforcement in the prism and in the slab was placed so as to make the specimen as much like the corresponding section of the ship as possible. The three bars placed at the junction of the prism and the slab made the section weak in resistance to a tensile stress tending to separate the slab from the rib. The specimens were designated by letters from A to L, inclusive.

Three types of stirrups were used, as shown in Fig. 71, and designated as Types I, II, and III, respectively. The stirrups were made from one-half-inch plain round steel bars, bent into U shape with hooked ends. Type I stirrups had the hooked ends

bent perpendicular to the plane of the legs and were used in specimens D, E, F, K, and L. Type II stirrups had the ends bent into loops through an angle of 270°, in which the planes of the loops were perpendicular to the plane of the legs. Stirrups of Type II were used in specimens A, B, C, G, H, and I. Type III stirrups had the ends bent outward at 90° from the legs and in the same plane. This type was used in specimen J.

Two stirrups of the same type were used in each specimen. They were placed 6 inches apart. The hooked ends of the stirrups were arranged at different depths in the slab member of the specimens and, in consequence, varied in relation to the position of the slab reinforcement. The distance between the hooks and lower surface of the slab for each specimen is shown in Table 15. The loops in the stirrups used in specimens G, H, and I were

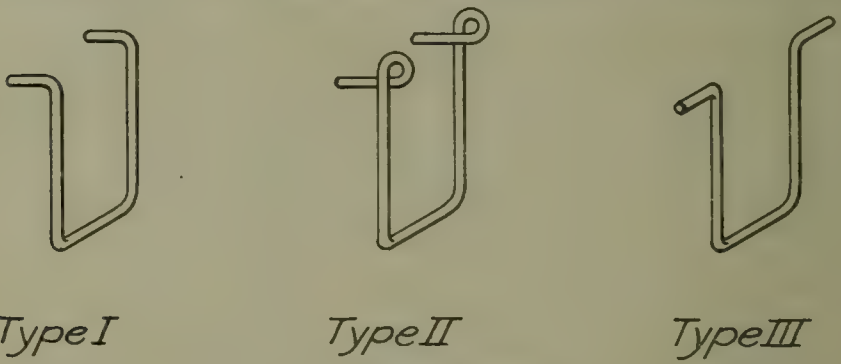


FIG. 71.—*Types of stirrups*

stuffed with mineral wool in order to exclude concrete during the pouring and to produce the condition of a void within the loop, a condition which is sometimes found in actual construction.

Fig. 72 shows the elevation and cross section of a typical specimen with the reinforcement indicated.

3. MAKING OF SPECIMENS

The concrete was mixed in the proportions 1:⅔:1⅓, measured by volume. Lehigh Portland cement and burnt-clay aggregates manufactured by the Atlas Portland Cement Co. were used. The consistency of the concrete was determined for each batch by the slump test; the average drop in a 6 by 12 inch cylinder was 9 inches when the mold was lifted. The concrete was representative of that used in the construction of concrete ships. In specimens A to F, inclusive, it was necessary to substitute sand and gravel

in the concrete used to a depth of about 6 inches in the rib of the specimen. This change was required on account of a shortage in the amount of burnt-clay aggregates available. The results of the tests show no effect that may be attributed to this change in mix. The specimens were poured from the top of the slab after the reinforcement was wired in position.

4. AUXILIARY SPECIMENS

To determine the strength of the concrete at the same ages as the stirrup pull-out specimens when they were tested, 19 control cylinders, 3 by 6 inches in size, were molded from the batches mixed for the pull-out specimens. The average results of the

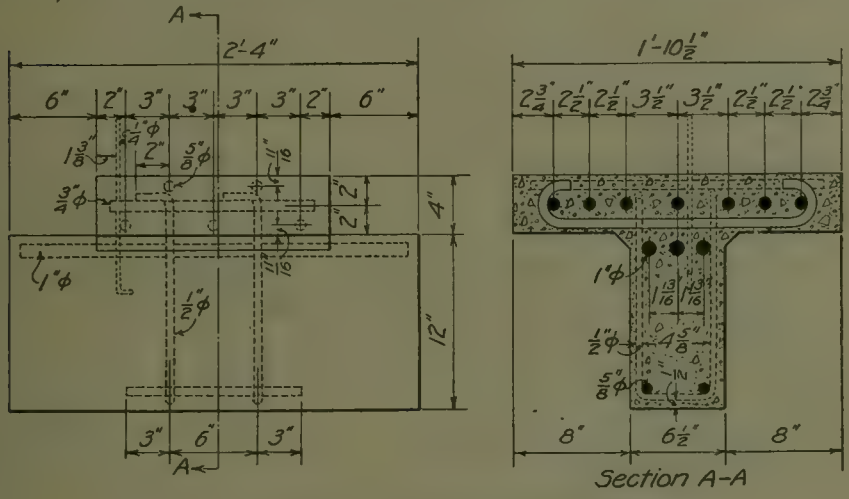


FIG. 72.—*Details of stirrup pull-out specimen*

compression tests on these cylinders are given in Table 15 and show a range of strength from 3900 to 4900 pounds per square inch.

5. METHOD OF TESTING

The stirrup pull-out specimens were tested in an Olsen testing machine of 200 000 pounds capacity. Fig. 73 shows a specimen arranged for testing.

A one-fourth inch plain round bar extended vertically through the slab of each specimen and 4 inches down into the rib. The bar was located 1⅜ inches from one edge of the slab. The lower end was anchored in the rib by means of a short L hook; the other end projected 4 inches above the slab. The upper half of the embedded length, or that passing through the slab member,

was coated with cup grease to destroy the bond resistance between the concrete and the rod. The purpose of this rod was to hold an Ames dial gage in contact with the slab during the test in order to determine, if possible, the instant when separation between the slab and the rib started.

The manner of loading the specimen was such as to pull the slab away from the rib. The overhanging ends of the slab member were supported on bearing plates and steel rollers by I beams, which were laid on the weighing table of the testing machine. The load was applied by the downward movement of the pulling head of the machine acting through a steel beam and spherical

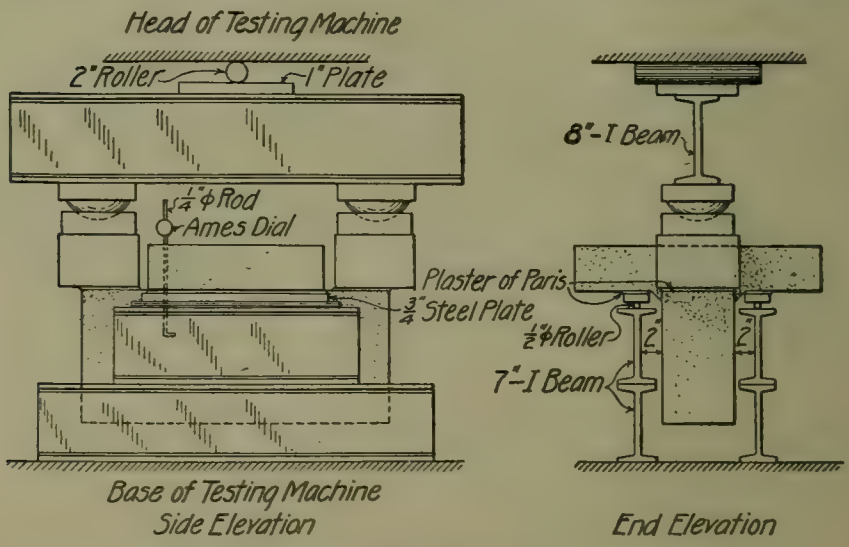


FIG. 73.—*Arrangement for testing stirrup pull-out specimen*

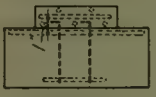
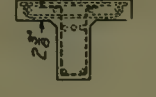
bearing blocks on the ends of the rib member of the specimen. The machine was stopped for examination of the specimen after each 3000-pound increment of load was applied.

The first indication of impending failure came by the sudden appearance of cracks in the plane at the junction of the slab and the rib of the specimen. Use of the Ames gage was abandoned after testing two or three specimens, as it gave no indications of value prior to failure.

6. RESULTS OF TESTS

Table 15 shows that the first crack occurred at about the same total load in all of the specimens except B and C, in which the load was somewhat higher. The sketches in Table 15 show that

TABLE 15.—Tests of Stirrup Pull-Out Specimens

Specimen and type	Age	Compressive strength of concrete	Load at first crack	Computed tensile stress in stirrups at first crack	Maximum load	Computed tensile stress in stirrups at maximum load	View showing position of stirrups	End view showing first crack
	Days.	Lbs./in.²	Pounds.	Lbs./in.²	Pounds.	Lbs./in.²		
A–II.....	31	4000	14 000	17 800	22 600	28 800		
B–II.....	[illegible]	[illegible]	19 000	24 200	23 500	29 900		
C–II.....	[illegible]	4000	18 000	22 900	32 700	41 600		
D–I.....	32	4100	14 000	17 800	15 300	19 500		
E–I.....	31	4000	12 500	15 900	12 500	15 900		
F–I.....	30	[illegible]	15 000	19 100	19 500	24 800		
G–II.....	17	[illegible]	15 000	19 100	21 700	27 600		
H–II.....	[illegible]	[illegible]	13 500	17 200	19 000	24 200		
I–II.....	12	4500	15 000	19 100	18 500	23 600		
J–I.....	14	4600	16 500	21 000	25 000	31 800		
K–I.....	17	[illegible]	14 000	17 800	16 000	20 400		
L–III....	13	[illegible]	12 000	15 300	19 000	24 200		

for most of the specimens the surface of rupture was substantially a plane coinciding with the lower surface of the slab. They indicate that for specimens B and C the surface of rupture had an area somewhat greater than that for the other specimens. If this could be taken into account in the calculations, the tensile stress at the time of the formation of the first crack might be found to be nearly the same for all specimens. Immediately after the failure of the concrete the entire load on the specimen was transferred to the stirrups. The position of the surface of rupture caused by the first crack had an important bearing upon the total resistance of the stirrup anchorage, since in many cases it passed so close to the hooks as to leave them little covering of concrete.

The tests show that Type II stirrups were much more effective when the loops were filled with concrete than when they were empty, and under such conditions were stronger than either Type I or Type III. Even when the loops were not filled with concrete the stirrups of Type II were better on the average than those of Type I. The one specimen having Type III stirrups gave greater load at the first crack and greater maximum load than any of the specimens having stirrups of Type I. The first crack probably had less effect on the embedment of the hooks in the Type III stirrups than in the stirrups of Type I.

In general, the stirrups which had the deepest embedment of the hooked and looped ends showed the greatest strength. An embedment of not less than 2 inches is apparently desirable.

Comparison of specimen D with E and G with H indicates that no increase in effectiveness was obtained by having the stirrup hooks rest on a bar of steel in the slab reinforcement.

In making comparisons consideration should be given to the difference in age of the specimens when tested.

The appearance of the specimens after failure is shown in Figs. 74 to 85, inclusive.

WASHINGTON, April 28, 1920.

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FIG. 74.—*View of specimen A after failure*

FIG. 75.—*View of specimen D after failure*

FIG. 76.—*View of specimen B after failure*

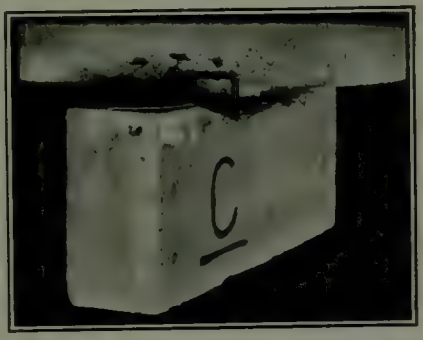

FIG. 77.—*View of specimen C after failure*

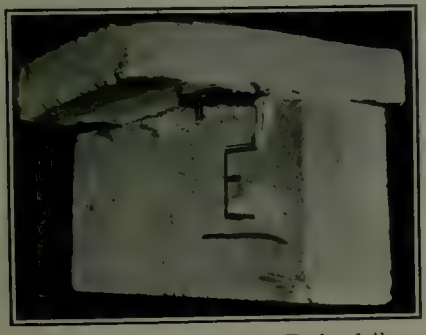

FIG. 78.—*View of specimen E after failure*

FIG. 79.—*View of specimen F after failure*

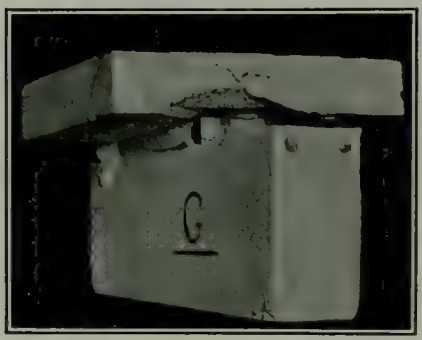

FIG. 80.—*View of specimen G after failure*

FIG. 81.—*View of specimen H after failure*

FIG. 82.—*View of specimen I after failure*

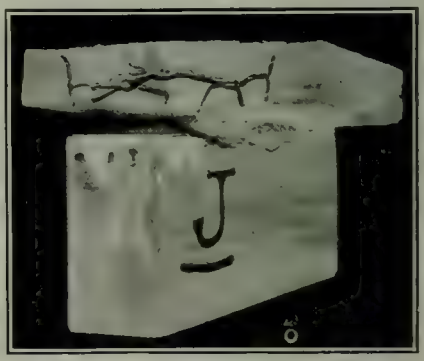

FIG. 83.—*View of specimen J after failure*

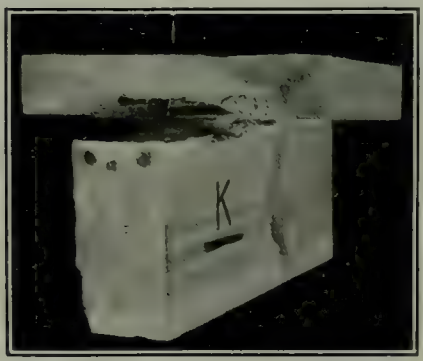

FIG. 84.—*View of specimen K after failure*

FIG. 85.—*View of specimen L after failure*

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No. 174

EFFECTS OF CAL AS AN ACCELERATOR OF THE HARDENING OF PORTLAND CEMENT MIXTURES

BY

ROY N. YOUNG, Associate Chemical Engineer
Bureau of Standards

OCTOBER 11, 1920

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EFFECTS OF CAL AS AN ACCELERATOR OF THE HARDENING OF PORTLAND CEMENT MIXTURES[1]

By Roy N. Young

CONTENTS

	Page
I. Introduction	4
II. Nature of Cal	5
III. Effect of Cal on the setting time of Portland cement	7
IV. Effect of Cal on the soundness of Portland cement	8
V. Effect of Cal on the early compressive strength of Portland cement mortars	9
1. Water storage	9
2. Air storage	10
3. Cold storage	10
4. Mixtures of cement and Cal stored in air	11
VI. Efflorescence	12
VII. Effect of Cal on steel reinforcing bars	12
VIII. Effects of Cal and calcium chloride on concrete	13
1. Descriptions and tests of materials	13
(*a*) Cal	13
(*b*) Calcium chloride	13
(*c*) Cement	14
(*d*) Aggregate	15
2. Methods and apparatus	15
(*a*) Proportioning	15
(*b*) Consistency	15
(*c*) Mixing	16
(*d*) Molding and capping	17
(*e*) Storage	17
(*f*) Testing	17
3. Results of strength tests	18
IX. Effect of Cal on consistency and workability of cement mixtures	21
X. Summary	22

[1] As explained in the introduction, the effect of calcium chloride in Portland cement mixtures has been quite thoroughly investigated, and the use of hydrated lime in such mixtures is not uncommon in practice. There are no restrictions on the use of these materials in concrete, but their application in the form of calcium oxychloride, or "Cal," has been covered by patents. In view of the importance to users of cement and concrete of knowing the properties of this material and its value as an accelerator, particularly in comparison with calcium chloride, a cooperative investigation was undertaken by the Bureau of Standards and the patentee, the results of which are given in this paper.

I. INTRODUCTION

A demand for a practical and efficient material for accelerating the hardening of Portland cement mixtures has received considerable attention in recent years. The need for such a material becomes of more consequence with the introduction of more rapid methods in construction and with the increasing amount of concrete repair and replacement work. Serious problems arise when it becomes necessary to interrupt or divert traffic for the duration of time required for concrete to harden sufficiently. The increased cost of lumber for forms has necessitated a more judicious use of such material, and its removal for further use as soon as the strength of the concrete will allow. This applies even more to the use of steel forms, which are rapidly replacing wooden forms in a great many types of concrete construction.

Considerable work toward the development of an accelerator for concrete has been done by the U. S. Bureau of Standards,[2] which found that calcium chloride was the most effective of the various substances tested. Comprehensive tests were made on concrete gaged with solutions up to 10 per cent commercial calcium chloride for the purpose of substantiating the results of the preliminary tests and also to determine if any injurious effects would result at later periods from the use of this accelerator. The increase in strength obtained at the age of two or three days over that of similar concrete gaged with water was from 30 to 100 per cent.[3] At the age of one year there was no indication that the addition of this salt had any deleterious effect on the durability of concrete. The three-year test pieces are now being broken with results showing further evidence that calcium chloride does not effect the ultimate integrity of concrete.

Inasmuch as the use of commercial calcium chloride in concrete is attended by difficulties caused by its highly hygroscopic property and by the handling of the solution, Charles Catlett[4] conceived the idea of introducing the salt by means of Cal, a material by which the above difficulties might be eliminated. Furthermore, Cal possesses possibilities which calcium chloride does not have, owing to the fact that the products of its decomposition by water are calcium chloride and calcium hydroxide, equivalent in effect to a simultaneous addition of calcium chloride and finely divided hydrated lime. Since both calcium

[2] Engineering Record, 74, pp. 266–267; 1916.
[3] Annual Report of the Director of the Bureau of Standards; 1918.
[4] Patent No. 1282188; patent No. 1308932.

chloride and hydrated lime are being used separately in cement mixtures to produce certain desirable effects, it was not considered that the use of Cal introduces anything new in the making of concrete, excepting the manner in which these materials are added.

Influenced by the above considerations, an investigation was inaugurated by Mr. Catlett, with the cooperation of the Bureau of Standards, for the purpose of studying the effects of Cal on Portland cement mixtures. The most important object was to learn the value of this material as an accelerator of the hardening of concrete.

While a greater part of the work preliminary to the main series of concrete tests deals with the effects of Cal on the early time strengths of cement mixtures under various conditions, it also includes miscellaneous tests bearing on questions which might preclude the use of this material with Portland cement, or limit its use to special mixtures or kinds of construction work.

Acknowledgment is made to G. M. Williams for assistance in planning the concrete tests, to Watson Davis for assistance in conducting these tests, and to A. B. Peck for the microscopic work.

II. NATURE OF CAL

Cal is essentially an oxychloride of calcium. A number of investigators have prepared crystals of this salt by boiling calcium hydroxide in a fairly strong solution of calcium chloride, filtering rapidly, and allowing the filtrate to cool. An analysis by Beesley [5] indicated that the formula was $3CaO.CaCl_2$, $14H_2O$. According to Rose [6] and André,[7] the crystals have the formula $3CaO.CaCl_2$, $16H_2O$. Grimshaw [8] found the constituents in the ratio $3CaO.CaCl_2$, $15H_2O$, but considered the formula $ClCa.O.Ca(OH)$, $7H_2O$ to be simpler and more intelligible.

Calcium oxychloride is readily soluble in water, in which it decomposes into calcium hydroxide and calcium chloride. It is also soluble in alcohol. The salt is acted upon by carbon dioxide, forming calcium carbonate and calcium chloride. Over sulphuric acid, in a vacuum or in the air, it parts with a portion of the water of crystallization. The crystals are needle-shaped and were

[5] Pharmaceutical Journal, 9, p. 568.
[6] Schweigger's Journal für Chemie und Physik, 5, pp. 29–155.
[7] Comptes Rendus, 92, p. 1452.
[8] Chemical News, 30, p. 280.

prepared by the writer from very minute size up to 1¼ inches in length, depending largely upon the rate at which the solution was cooled. The composition of the crystals made under various conditions was in all cases the same and agreed closely with the formula given by Grimshaw.

Cal is made by pulverizing the product resulting from a mixture of lime, calcium chloride, and water. The material used in the following tests was made by the Mellon Institute at Pittsburgh, Pa., in September, 1918, by mixing together 100 parts hydrated lime, 55 parts commercial calcium chloride, and 50 parts water. During several weeks exposure to the atmosphere it became hard and dry.

There was a question as to whether this material was chiefly a mechanical mixture or a product of chemical reaction. A microscopic examination was made, and it was found to consist of fairly well developed crystals of calcium oxychloride lying in a groundmass of minute irregular crystals which resembled the larger ones in every other respect. There was only a small percentage of calcium hydroxide present. Other samples were prepared by treating separately quicklime and hydrated lime with various concentrations of calcium chloride solutions. These proved to be of the same nature as the Cal just described, with the exception that the larger crystals varied in size and relative number among the different samples.

The first lot of Cal was ground without further drying than that which it received by exposure to the atmosphere. The finished product was a dry, white, rather fluffy powder which analyzed as follows:[9]

	Per cent
Loss on ignition	43.93
Hydrochloric acid insoluble	.15
SiO_2	.71
R_2O_3	.51
CaO	41.15
MgO	3.58
Cl_2	11.47
SO_3	.71
	102.21
O=C=Cl	2.59
Total	99.62
CO_2	3.78
Water (loss—CO_2)	40.15

[9] The Cal used in the concrete tests was dried and will be described later.

If Cal is exposed to the air, it gradually takes up carbon dioxide and becomes damp. However this action is comparatively slow and should not cause trouble if the material is handled in the same way as hydrated lime.

A sample of Cal which had been exposed to the laboratory air for eight months and another which had been stored in a cloth bag for about three years were used for treating mortars in compression tests. The results indicated that exposure does not lessen the effectiveness of Cal as an accelerator of the hardening of Portland cement mixtures.

III. EFFECT OF CAL ON THE SETTING TIME OF PORTLAND CEMENT

The effect of Cal on the setting time of normal Portland cements may be seen by examining the results given below. The set was hastened in all cases and to a greater extent by the larger percentage of Cal.

TABLE 1.—The Setting Time of Normal Portland Cement Treated with Undried Cal

[The percentage of water is calculated on the basis of the combined weights of the cement and Cal]

Cement	Ratio (percentage) Cal to cement	Ratio (percentage) water to total mixture	Initial set	Final set
Sample Nos.:			H. M.	H. M.
1	None	23.4	4 0	8 10
	5	21.9	2 15	5 45
	8	21.0	1 50	5 30
2	None	23.2	3 35	6 45
	5	21.7	1 50	4 10
	8	[illegible]	1 25	3 30
3	None	23.4	3 40	7 0
	5	21.9	2 45	5 45
	8	21.0	2 5	4 25
4	None	24.0	4 40	7 30
	5	22.5	3 0	5 40
	8	21.6	1 50	3 40
5	None	22.5	3 35	6 25
	5	21.0	1 55	2 55
	8	20.1	1 5	1 55

It will be noticed that the percentage of water necessary for normal consistency decreases as the amount of Cal increases. This may be explained in part by the fact that the Cal contains considerable water which is set free upon its decomposition and which is in excess of that necessary to give plasticity to the resulting products.

Cal was added to a number ot quick-setting cements with the result that they were made normal. This may seem contradictory to the results obtained with normal cements as shown above, but it should be remembered that the setting time in such cases is not controlled by Cal alone. Ground Portland cement clinker is usually quick setting, and therefore gypsum is added as a retarder in order to meet commercial requirements. If Cal is added to a finished cement, the effect on the set will be the resultant effect of both Cal and gypsum. The effect of Cal alone on ground cement clinker is as follows:

TABLE 2.—The Setting Time of Ground Cement Clinker Treated with Cal

Cal	Ratio (percentage) water to mixture	Initial set	Final set
		H. M.	H. M.
None	25.0	Flash ..	.....
2 per cent	24.5	1 10	5 0
4 per cent	23.1	3 10	6 10
6 per cent	21.7	2 55	5 45
8 per cent	21.3	2 30	4 20
10 per cent	20.0	1 10	3 25

It will be seen that the maximum retardation is produced by 4 per cent of Cal, and that the retardation diminishes with increasing or decreasing amounts. Hence it seems reasonable that an addition of Cal to a normal cement already containing gypsum should hasten the set in the same way that an addition of Cal to the clinker already containing 4 per cent of Cal will hasten the set. Also, if a cement containing gypsum is quick setting, an addition of Cal in increasing amounts should increase the retardation until a maximum retardation is reached due to the combined effect of Cal and gypsum.

IV. EFFECT OF CAL ON THE SOUNDNESS OF PORTLAND CEMENT

No indication of unsoundness has appeared in normal cement treated with Cal. Unsoundness, as determined by the steam test, may be corrected by the addition of Cal, according to the results of tests given below. This action is probably due to an increased hydration of the free lime before the cement sets.

A very unsound cement was used in a 1:3 mortar which was molded into 2-inch cubes and stored in air after 24 hours. At the end of 8 months, the plain mortar test pieces were entirely

disintegrated. The test pieces made from the same mortar to which 5 per cent of undried Cal was added were still very firm, although a few disintegration cracks had developed.

TABLE 3.—Effect of Cal on the Soundness of Portland Cement

Cement laboratory No.	Soundness after addition of various percentages of undried Cal			
	0 per cent	2 per cent	5 per cent	8 per cent
42895	Unsound; slightly warped, not hard.	Sound; very slightly warped.	Sound; very slightly warped.	Sound.
42896	do	Unsound; warped, hard.	Sound; very slightly warped, hard.	Sound; very slightly warped, hard.
43015	Unsound; very soft	Unsound; warped, not hard.	Sound	Sound.
43200	do	Sound	do	Do.
5	do		Sound; slightly warped, hard.	Do.

V. EFFECT OF CAL ON THE EARLY COMPRESSIVE STRENGTH OF PORTLAND CEMENT MORTARS

1. WATER STORAGE

A series of tests was made on five different brands of cement, using 1 part cement to 3 parts Standard Ottawa sand. Per centages of undried Cal varying from 0 per cent to 8 per cent of the cement were added to the dry mixtures. Also, mortars were made from each brand of cement by gaging the mixtures with a 4 per cent solution of commercial calcium chloride. This concentration was used for the reason that it was found to be the most satisfactory in tests previously conducted by this Bureau.

Mixes containing additions of Cal up to 5 per cent were, in some cases, gaged with the same quantity of water as was used in the plain mortar for normal consistency. Consequently the excess of water contained in the Cal produced a wetter consistency than normal, and probably resulted in somewhat smaller increases in strength over the plain mortars than would have been obtained if the consistencies had been the same throughout. All other mixes were of normal consistency as calculated from the normal consistencies of the neat mixtures, or as nearly as could be judged by the operators. The results of these tests are shown in Fig. 1. In general, with each increase in the amount of Cal added to the mortar there is an increase in strength at each of the three periods tested.

2. AIR STORAGE

Some of the above tests were repeated, with the exception that the test pieces were stored in the laboratory air rather than in water. The results of these tests are shown in Fig. 2. It is evident from the much lower strengths of the 28-day test pieces stored in air that there was a very rapid drying out of these test pieces, as might be expected on account of their size. The effect of Cal under the conditions of these tests is remarkable.

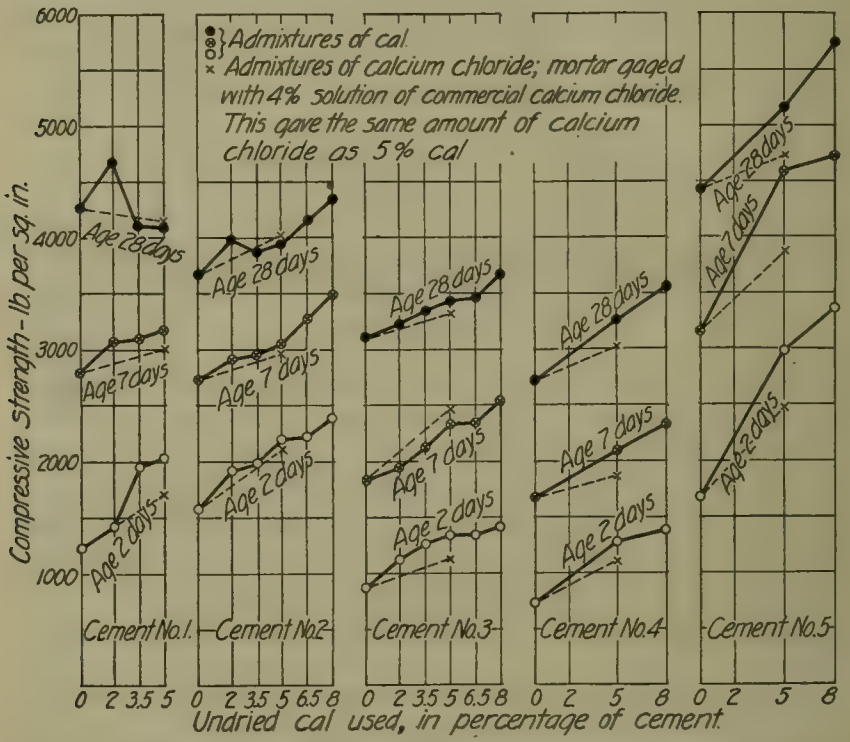

FIG. 1.—*Compressive strength of mortar at various ages treated with Cal or calcium chloride and stored in water*

The strength of the treated mortar at 2 days is considerably above that of the untreated mortar at 28 days.

3. COLD STORAGE

It is a well-known fact that the rate of hardening of cement is affected by the temperature conditions. One difficulty arising from this is slow setting and hardening, which often occurs during cold weather. Tests in which the test pieces were stored in a refrigerator at a temperature slightly above freezing indicate

that such difficulties may be overcome by the use of Cal. Also, Cal may be a means of protection against freezing in that it greatly reduces the critical time during which freezing is injurious.

4. MIXTURES OF CEMENT AND CAL STORED IN AIR

As it might be desirable to mix an accelerator with cement at the mill during the process of grinding or before it is packed, a series of tests was made to determine the effects of air storage on mixtures of cement and Cal. The mixtures in these tests were portions of the ones from which the mortar tests shown in Fig. 1 were made. About 2 kg of each mixture were placed in

FIG. 2.—*Compressive strength of mortar at various ages treated with Cal and stored in air*

a small canvas bag and stored in the laboratory air. At the end of 90 days mortar cubes were made under as nearly as possible the same conditions as those existing during the former tests.

All samples of cement and mixtures of cement and Cal deteriorated during the storage period. There was a tendency for the mixtures to form lumps and pack. The mortar tests showed no marked increase in strength of mortar containing Cal over that of the untreated. After these tests were started, it was found that the Cal used gives up considerable moisture when exposed to the air, so no doubt that in this instance the moisture given up by the Cal which was mixed with the cement did much toward affecting this deterioration. It was not determined whether the result might be entirely attributed to this loosely

held water or whether the Cal increased the amount of moisture taken up by the cement from the air. It may be possible that a change in the method of manufacture of Cal will produce a material which can be mixed with cement without ill effects and at the same time retain its efficiency when the mixture is used in concrete.

VI. EFFLORESCENCE

Two small brick walls were built on the roof of a building where they were exposed to the weather. Plain mortar (1 part cement to 3 parts Potomac River sand, by weight) was used in constructing one of the walls, while 8 per cent undried Cal was added to the mortar used in the other.

Considerable efflorescence was observed, but there was very little difference in the amounts on the two walls. There appeared to be slightly less on the one containing the Cal mortar. An analysis was made of the material which was caused to effloresce by artificial means from a mortar containing Cal. It consisted chiefly of the alkali carbonates and chlorides.

VII. EFFECT OF CAL ON STEEL REINFORCING BARS

One-half-inch steel reinforcing bars about 4½ inches long were embedded in 3 by 6 inch cylinders made of 1:3 standard Ottawa sand mortar. One set was made with no addition, one with an addition of 5 per cent undried Cal, and another was gaged with a solution of calcium chloride which furnished an amount of anhydrous calcium chloride equal to that contained in the 5 per cent addition of Cal. These cylinders were stored exposed to the weather. At the end of eight months one test piece from each set was broken, and the only signs of corrosion found were a few rust spots on the bar embedded in mortar containing calcium chloride. Of course, these tests furnish no conclusive information, but it is interesting to note that corrosion had not appeared on the bar in the mortar containing Cal. Such tests should be continued for a number of years and under various moisture conditions.

VIII. EFFECTS OF CAL AND CALCIUM CHLORIDE ON CONCRETE

1. DESCRIPTIONS AND TESTS OF MATERIALS

(a) CAL

It was found advantageous to drive off a portion of the water in the Cal, and therefore in preparing the lot used in the following tests the lump material was dried. This was done in such a way that it was not exposed to the atmosphere or gases from the burner during the operation. The crushing was done in a small jaw crusher and the grinding in a pebble mill. A sample of about 100 pounds was mixed thoroughly by placing it in the pebble mill lightly loaded with pebbles. The entire quantity was quartered down for the chemical sample. An anlysis of this sample was made several days before the tests were started, and the chlorine content was again determined after all the concrete was mixed, to learn if any change in the percentage, caused by exposure to the air, had taken place during this time. It may be seen by the results given below that equal weights of the material taken at any time during the tests furnished practically equal amounts of calcium chloride.

CHEMICAL ANALYSIS OF CAL

[Laboratory No. 56971]

	Per cent
Loss on ignition	21.42
Insoluble material	.80
Soluble material:	
SiO_2	1.00
R_2O_3	.60
CaO	58.10
MgO	4.75
Cl_2	15.68
SO_3	.91
	103.26
O=Cl	3.54
Total	99.72
CO_2	5.12
Water (loss—CO_2)	16.30
Chlorine content after about eight weeks	15.50

(b) CALCIUM CHLORIDE

A solution of calcium chloride was made from commercial calcium chloride and filtered through a finely woven cloth, separating considerable insoluble matter. After a sample was taken for chemical analysis, the solution was kept in an air-tight container

until used. The samples for chemical analysis of this calcium chloride solution were taken at the same time as those for the analysis of Cal. The analysis of this solution follows:

RESULTS OF ANALYSIS OF CALCIUM CHLORIDE SOLUTION

[Specific gravity at 24° C=1.338]

	Per cent
Ca	12.16
Cl_2	22.31
Mg, less than	.10
Chlorine content after the concrete mixing was completed	22.42

(*c*) CEMENT

On account of the somewhat varying effectiveness of Cal on different brands of cement as indicated by the mortar tests, it was decided that no less than three brands should be used in this series. These brands were chosen from those used in the mortar tests for the reason that they had been found to fairly well represent the range of Portland cements with regard to early time strengths and that they could be purchased on the market at Washington, D. C.

The entire lot of each brand was thoroughly mixed on canvas and then placed in metal containers until used. The chemical and physical tests of these cements are given in Table 4.

TABLE 4.—Chemical and Physical Tests of Cements

Nature of determination	Test results for laboratory numbers—		
	45001	45076	45101
Chemical:	Per cent	Per cent	Per cent
Loss	2.01	2.43	1.76
Insoluble residue	.20	.17	.40
Silica	19.78	22.39	20.72
Oxide of iron	3.40	2.20	1.90
Oxide of alumina	7.26	5.58	5.82
Lime	62.56	60.00	63.48
Magnesia	1.72	4.07	4.02
Sulphuric anhydride	1.91	1.78	1.63
Sodium oxide	.....	.87	.43
Potassium oxide	.....	.42	.38
Physical:			
Water used—			
Neat	23.0	23.0	23.0
Mortar	10.3	10.3	10.3
Fineness (200-mesh sieve)	84.5	81.6	87.1
Time of set—	Hr. Min.	Hr. Min.	Hr. Min.
Initial	2 55	2 55	3 45
Final	6 30	6 00	6 55
Soundness	O. K.	O. K.	O. K.
Tensile strength—	Lbs./in.²	Lbs./in.²	Lbs./in.²
7 days	283	200	318
28 days	370	313	455

(*d*) AGGREGATE

Potomac River aggregate was used in this series and was purchased in 5-ton lots at Washington, D. C. The gravel was washed, dried, and stored in sacks until used. To insure uniformity, just before each day's run of mixing, a sufficient amount of gravel for all mixes for one proportion and for one brand of cement was mixed by shovel on a concrete floor and resacked in lots of accurate weight for one batch. The sand was treated in the same manner excepting that it was not washed, as it was in good condition when received. The results of the physical tests of these materials are given below. The weights per cubic foot used in proportioning were 95 per cent of the weight per cubic foot of packed material. A metal cubic-foot measure was used in this determination.

WEIGHTS PER CUBIC FOOT

	Pounds
Sand	120
Gravel	114

TABLE 5.—Mechanical Sieve Analysis of Aggregate

Sieve No.	Size opening	Per cent passing		Sieve No.	Size opening	Per cent passing	
		Sand	Gravel			Sand	Gravel
	Inches				Inch		
	1½		100	4	0.185	98	1
	1		91	8	.093	81	
	¾		71	14	.046	69	
	½		51	28	.0232	51	
	⅜		26	48	.0116	15	
3	.263	100	1	100	[illegible]	4	

2. METHODS AND APPARATUS

(*a*) PROPORTIONING

The proportioning of the cement, sand, and gravel was by volume, but for the sake of convenience and accuracy the materials were measured by weight. The cement was assumed to weigh 94 pounds per cubic foot.

In all mixes in which a direct comparison was made between Cal and calcium chloride, an exact weight of the calcium chloride solution was used which contained an equivalent weight of chlorine as added by means of the Cal.

(*b*) CONSISTENCY

The consistency of the various mixes was measured by means of the "flow table," which has been developed by the Bureau of Standards.[10] It is believed that this method is the most satis-

[10] Williams, Engineering News Record, 84, p. 1044.

factory of any so far devised for determining the relative "flowability" of concrete. The method is sensitive through a wider range of consistencies than the slump method, and the results are consistent and reliable.

Two consistencies were chosen as standards in the concrete tests. The 1 : 1½ : 3 concrete was rather stiff and represented the type of concrete often used in road construction. All batches of this proportion were made to have a relative flowability as near 150 as possible.

The 1 : 2 : 4 concrete was considerably wetter and was of such consistency as is often used in reinforced-concrete construction. The relative flowability sought in this case was 200.

From two to three flow tests were made on each batch in order that very nearly the desired consistency might be produced before the final test was made. This final test was to be made 10 minutes after the water was added to the dry mixtures.

(c) MIXING

In the preliminary tests on concrete contradictory results were obtained under certain conditions in that the strengths of the treated concrete at seven days were lower than the corresponding strengths of the untreated concrete. Considerable work was done to determine the cause of this before it became evident that the different method used in mixing the concrete was the source of the difficulty. In every instance the mortars were mixed by kneading with the hands, and the batches were sufficiently small to allow thorough mixing within one and one-half minutes. The batches of concrete were considerably larger and were mixed by turning with a trowel. Consequently they did not receive as vigorous or thorough mixing as applied to the mortars. When these concrete mixes were duplicated and mixed by machine the results were of the same order as those obtained in the mortar tests. No further investigation was made in this direction, but it is believed that the results obtained with the hand-mixed concrete were exceptional.

In the following tests the mixing was done in a model 15 Wonder mixer. Sufficient material was used in each batch to provide some in excess of the amount necessary for six 6 by 12 inch cylinder test pieces. After the dry materials were placed in the mixer a part of the water was added and the mixer was started. More water was then added until about the proper consistency was obtained. At the end of 2 minutes from the time the first water

was added the mixer was stopped and a "flow" test was made. When only two flow tests were made within 10 minutes after the water was added the mixer was run 45 seconds just previous to the making of the final flow test. Later three flow tests were made instead of two, and the mixer was run for 30 seconds before each of the last two tests. This additional mixing of all batches was necessary in order that water might be added if needed to increase the flowability to the desired degree and at the same time that all batches might be given an equal amount of mixing.

(*d*) MOLDING AND CAPPING

All test pieces in this series were 6 by 12 inch cylinders. The molds used were made from wrought-iron pipe cut in lengths of 12 inches and slit perpendicularly to the axis of the cylinder. This slit was kept closed during molding by bolts through angles riveted to the cylinder on each side of the slit. The molds were filled in three layers, each layer being puddled with a five-eighth-inch steel rod. The bottom caps were formed by placing the molds on a layer of stiff neat cement which was spread about one-half inch thick on sheet iron a short time before molding the test pieces. The molds were filled to less than one-fourth inch from the top. After about an hour the filling was completed with a thin neat cement paste and smoothed off with a trowel. This method gave good solid caps which required very little grinding to produce a plane bearing surface.

(*e*) STORAGE

At the end of 24 hours the test pieces were removed from the molds. Three of the test pieces from each batch were stored in damp sand, and the remaining three were stored in the air of the laboratory. Hence, the results obtained under the two storage conditions are strictly comparable with respect to materials, quantity of mixing water, etc. The daily average room temperatures where test pieces were stored in air gradually lowered throughout the storage period from about 72° F. to about 60° F. The temperature of the damp sand averaged about 5° F. below these temperatures at the beginning of the tests, but toward the end the temperatures of the two storages were about the same. The average relative humidity was approximately 65 per cent.

(*f*) TESTING

Specimens 62–1–1 to 62–3–3 and 62–13–1 to 62–15–3,[11] inclusive, were tested on a 200 000-pound Riehle testing machine. All others

[11] The numbering system is merely a local means of identifying the test pieces.

were tested on a 100 000-pound Riehle machine. A single thickness of blotting paper was placed between each end of the specimen and the bearing plate. The test piece was centered on a spherical bearing block which was then shifted into place. The upper surface of the test piece was fitted squarely against the upper bearing surface by turning the test piece and upper part of the block horizontally first in one direction and then in the other while the movable head was being lowered onto the test piece.

3. RESULTS OF STRENGTH TESTS

The results of the strength tests are given in Table 7.

The most important facts deduced from these data are as follows:

1. In all cases, the strength of the treated concrete was greater than that of the corresponding untreated concrete at the two and seven day periods.

2. The strength of the treated concrete was practically the same as that of the untreated at the 28-day period.

3. The effect of Cal at the 2-day period was very nearly the same for all cements, proportions, and conditions of the tests. This may be seen by examining the data given below.

TABLE 6.—Percentage Increase in Strength of Concrete Due to Cal at the Two-Day Period

Resulting from—	Maximum	Minimum	Average
Cement No. 45003	75	50	66
Cement No. 45076	80	55	65
Cement No. 45101	66	52	58
Proportion 1 : 1½ : 3, stiff consistency	61	52	58
Proportion 1 : 2 : 4, wet consistency	80	52	68
Stored in damp sand	80	52	65
Stored in air	69	52	61

4. The results produced by Cal and calcium chloride are practically the same.

Since the primary reason for using an accelerator in Portland cement mixtures is to produce a given strength in less time than ordinarily required and thereby effect a saving, it is important to express laboratory tests on this basis. If time-strength curves of both the untreated and corresponding treated mixtures are plotted together, it is very convenient to compare the ages at which they have equal strengths. In this series of concrete tests, the strength of the treated concrete at two days was equal to that of the untreated at from three to four and one-half days. Mortar tests indicated that more marked results may be obtained by increasing the percentage of Cal.

Although very uniform results were obtained under various conditions at the 2-day period, there was considerable variation at the 28-day period. Furthermore, Cal generally increased the strength of mortars at the 28-day period, while it seemed to have little or no effect on the concrete at this age. A number of mortar tests were made with variations in proportions and percentages of Cal to cement and Cal to water, with the thought that they might determine the cause of these irregularities, but no conclusive results were obtained.

TABLE 7.—Compressive Strengths of Concrete to Which Cal and Calcium Chloride Have Been Added

[Additions of Cal throughout this series were 5 per cent of the cement. Cal and calcium chloride additions contained equal amounts of chlorine. Test pieces were 6 by 12 inch cylinders]

CEMENT NO. 45003

Batch	Addition	Proportion	Relative flowability	Water, per cent	Compressive strength in pounds per square inch					
					Stored in damp sand			Stored in air		
					2 days	7 days	28 days	2 days	7 days	28 days
62–1–1	None	1 : 1½ : 3	150	7.88	870	2390	3491	806	2370	3662
2	do	1 : 1½ : 3	156	7.88	915	2390	4035	882	2450	3851
3	do	1 : 1½ : 3	158	7.88	975	2290	3735	918	2190	3508
Average			155	7.88	900	2337	3754	868	2337	3507
62–2–1	Cal	1 : 1½ : 3	143	7.88	1488	3030	3975	1290	2470	3379
2	do	1 : 1½ : 3	148	7.88	1432	2770	3768	1536	2618	3500
3	do	1 : 1½ : 3	149	7.88	1435	2650	3687	1370	3000	3689
Average			149	7.88	1450	2817	3617	1390	2693	3556
62–3–1	$CaCl_2$	1 : 1½ : 3	148	7.88	1686	2700	3471	1515	2610	3339
2	do	1 : 1½ : 3	149	7.88	1347	2790	3686	1340	2790	3665
3	do	1 : 1½ : 3	154	7.88	1600	2790	3390	1300	2790	3604
Average			152	7.88	1540	2760	3650	1450	2730	3536
62–4–1	None	1 : 2 : 4	180	8.52	338	1222	2212	322	1090	1782
2	do	1 : 2 : 4	212	8.52	341	1143	2314	357	1109	1947
3	do	1 : 2 : 4	201	8.52	184	1236	2303	392	1374	1978
Average			201	8.52	154	1262	2278	357	1191	1908
62–5–1	Cal	1 : 2 : 4	190	9.28	593	1329	1765	601	1242	1612
2	do	1 : 2 : 4	200	8.89	674	1340	1750	553	1295	1591
3	do	1 : 2 : 4	213	8.89	688	1455	1976	658	1338	1612
Average			201	9.02	618	1375	1830	603	1297	1605
62–6–1	$CaCl_2$	1 : 2 : 4	217	8.28	686	1450	2112	636	1439	2012
2	do	1 : 2 : 4	196	8.28	740	1885	1743	756	1562	1998
3	do	1 : 2 : 4	200	8.28	716	1515	2282	595	1491	1800
Average			206	8.28	714	1533	2112	676	1497	1937

TABLE 7.—Compressive Strengths of Concrete to Which Cal and Calcium Chloride Have Been Added—Continued

CEMENT NO. 45076

Batch	Addition	Proportion	Relative flowability	Water, per cent	Compressive strength in pounds per square inch					
					Stored in damp sand			Stored in air		
					2 days	7 days	28 days	2 days	7 days	28 days
62-7-1	None	1 : 1½ : 3	156	8.14	389	952	2362	396	1045	1733
2	do	1 : 1½ : 3	150	7.94	452	1141	2231	408	1117	1855
3	do	1 : 1½ : 3	151	7.94	428	1041	2327	395	1050	1822
Average			152	8.01	423	1045	2307	400	1071	1803
62-8-1	Cal	1 : 1½ : 3	147	8.21	585	1226	2228	621	1385	1922
2	do	1 : 1½ : 3	152	8.21	704	1183	2217	672	1363	2029
3	do	1 : 1½ : 3	142	8.21	538	1176	2257	643	1315	1883
Average			147	8.21	656	1196	2234	645	1354	1945
62-9-1	$CaCl_2$	1 : 1½ : 3	148	8.02	676	1329	2362	690	1400	2093
2	do	1 : 1½ : 3	155	8.02	651	1230	2292	690	1383	2030
3	do	1 : 1½ : 3	161	8.02	617	1277	2041	584	1403	1971
Average			155	8.02	651	1279	2232	658	1395	2031
62-10-1	None	1 : 2 : 4	188	8.80	132	393	1298	146	405	718
2	do	1 : 2 : 4	198	8.80	138	416	1145	141	393	647
3	do	1 : 2 : 4	205	8.80	113	346	1131	147	445	729
Average			197	8.80	128	385	1189	145	415	698
62-11-1	Cal	1 : 2 : 4	200	9.04	229	526	1146	228	516	873
2	do	1 : 2 : 4	210	9.04	252	539	1020	262	569	842
3	do	1 : 2 : 4	196	9.04	209	524	1223	230	633	920
Average			202	9.04	230	530	1130	240	573	878
62-12-1	$CaCl_2$	1 : 2 : 4	201	8.80	274	572	993	279	534	870
2	do	1 : 2 : 4	196	8.80	218	533	1141	251	634	893
3	do	1 : 2 : 4	201	8.80	238	544	1139	229	525	854
Average			199	8.80	243	550	1091	253	564	872

Cement No. 45101

62-13-1	None	1 : 1½ : 3	156	7.94	1146	2500	3660	1184	2375	3330
2	do	1 : 1½ : 3	149	7.75	1306	2742	4060	1175	2258	3350
3	do	1 : 1½ : 3	152	7.75	1212	2640	3900	1177	2347	3295
Average			152	7.81	1221	2761	3873	1179	2327	3325
62-14-1	Cal	1 : 1½ : 3	152	8.06	1875	3133	3905	1995	2970	3635
2	do	1 : 1½ : 3	150	8.06	1800	2955	3885	1801	2747	3525
3	do	1 : 1½ : 3	149	8.06	1887	3094	.....	1850	2631	3810
Average			150	8.06	1854	3027	3895	1882	2783	3657

TABLE 7.—Compressive Strengths of Concrete to Which Cal and Calcium Chloride Have Been Added—Continued

CEMENT NO. 45101—Continued

Batch	Addition	Proportion	Relative flowability	Water, per cent	Compressive strength in pounds per square inch					
					Stored in damp sand			Stored in air		
					2 days	7 days	28 days	2 days	2 days	28 days
62-15-1	$CaCl_2$	1 : 1½ : 3	149	7.75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4215
2	...do	1 : 1½ : 3	150	7.75	1835	[illegible]	4260	1973	[illegible]	3720
3	...do	1 : 1½ : 3	150	7.75	1943	[illegible]	3940	1775	[illegible]	3760
Average			150	7.75	1935	3034	4015	1910	2914	3898
62-19-1	None	1 : 2 : 4	[illegible]	8.67	535	1551	2757	457	1177	[illegible]
2	...do	1 : 2 : 4	199	[illegible]	504	1510	2422	524	1225	1965
3	...do	1 : 2 : 4	196	8.92	537	1475	2523	476	1145	1739
Average			[illegible]	8.66	525	1512	2567	[illegible]	1182	1837
62-20-1	Cal	1 : 2 : 4	[illegible]	[illegible]	912	.....	[illegible]	774	[illegible]	[illegible]
2	...do	1 : 2 : 4	194	[illegible]	862	1554	2418	738	[illegible]	1835
3	...do	1 : 2 : 4	[illegible]	8.68	[illegible]	1648	2240	702	1371	[illegible]
Average			196	8.68	870	1601	2380	[illegible]	1441	1982
62-21-1	$CaCl_2$	1 : 2 : 4	196	8.37	872	1694	[illegible]	868	1434	2162
2	...do	1 : 2 : 4	204	8.72	795	1782	[illegible]	784	1578	[illegible]
3	...do	1 : 2 : 4	196	8.56	754	1654	[illegible]	780	1458	[illegible]
Average			199	8.55	[illegible]	1710	2432	811	1490	1977

IX. EFFECT OF CAL ON CONSISTENCY AND WORKABILITY OF CEMENT MIXTURES

The way in which Cal affects consistency depends largely upon the amount of water which it contains. When Cal containing 40 per cent water was added to cement or mortar, an appreciably wetter mix was the result. Cal containing 16 per cent water had no effect on the consistency or flowability of mortars, but it appeared to increase the plasticity or ease in working the mixtures with a trowel. In the concrete tests a very small increase in percentage of water was necessary in the mixes containing Cal over that added to the untreated mixtures in order to produce the same relative flowability. This was probably due to the fact that Cal causes earlier stiffening or setting and that the flowability tests were made 10 minutes after the water was added. This earlier stiffening is not objectionable if the concrete is placed shortly after mixing. On the other hand, it is at times very desirable, especially in construction requiring a finished surface, such as floors.

Cal was used in a concrete floor topping alongside of topping containing no accelerator, for the purpose of observing the effect under actual field conditions. The treated topping was ready for the finishing operation in about one-half the time required by the untreated, as judged by the finisher. This agreed very closely with the results of the laboratory tests on the rate of setting of the two mixes by means of the flow table.

X. SUMMARY

Cal is a material obtained by pulverizing the dried or undried product resulting from a mixture of either quicklime or hydrated lime, calcium chloride, and water. It is a dry white powder which may be handled in much the same way as hydrated lime, and with the same general keeping qualities. It is much more convenient to handle and use in making concrete than calcium chloride, either fused or in concentrated solution. Upon exposure to the air Cal gradually takes up carbon dioxide and becomes somewhat damp. However, tests indicate that long exposure does not affect its action as an accelerator of the hardening of Portland cement mixtures.

The setting of normal Portland cement mixtures may be hastened by Cal to an extent which is very desirable in concrete construction requiring a finished surface. The finishing operation may proceed with much less delay after the concrete has been placed, which should result in cutting down overtime labor. This hastening of the set is not objectionable in any type of construction providing the concrete is placed soon after it is gaged with water. It is believed that Cal increases the workability of Portland cement mixtures. However, no attempt was made to measure the extent of this effect, because up to this time no satisfactory test has been developed for measuring the workability of Portland cement mixtures.

Unsound cements may be greatly benefited or made sound by an addition of Cal. This effect was produced in neat pats subjected to the steam test and in mortar test pieces stored in air.

Limited tests indicate that quicksetting cements, either fresh or having become quicksetting on aging, may be made normal by the addition of Cal as used in cement mixtures.

There was no indication that the amount of efflorescence appearing on the surface of cement mixtures exposed to the weather is increased by the use of Cal.

The series of tests on the effect of Cal on steel reinforcing bars which were embedded in a thin layer of Cal cement mortar and exposed to the weather for eight months without showing any sign of corrosion, while favorable, is too limited to give assurance that corrosion will not occur under these conditions. Caution should, therefore, be exercised in the use of Cal in concrete containing steel reinforcement when the concrete is to be freely exposed to the weather or excessive dampness. It is believed that no bad effect will be produced in ordinary building construction.

Undried Cal mixed with Portland cement causes greater deterioration in the quality of the cement during storage than that which ordinarily takes place. Therefore, Cal should be added to the concrete materials during the mixing operation, preferably before the water is added.

All Portland cement mixtures treated with Cal attained greater strength at the 2-day and 7-day periods than the corresponding untreated mixtures. The percentage increase in the strength of mortar at the 2-day period obtained by an addition of 5 per cent Cal to cement ranged from 40 to 140. The strength of the treated mortar at two days was equal to the strength of the untreated mortar at three and one-half to eight days. These calculations are made from the results of tests in which the test pieces were stored in water, damp sand, or damp closet.

Treated mortars stored in the laboratory air attained at 2 days strength greater than that of the untreated mortars at 28 days. This was due to the rapid drying out of the small test pieces and the comparatively slow rate of gain in strength after the 2-day period. However, this indicates that Cal is especially advantageous in cement mixtures which are necessarily subjected to any drying-out action.

The increase in strength produced by 5 per cent Cal in concrete mixtures at the 2-day period ranged from 52 to 135 per cent, and the strength of the treated concrete at the 2-day period was equal to that of the untreated at from three to four and one-half days. On an average this represents a saving of approximately one-half the time in operations which are dependent upon the strength of the concrete at early periods. The effect of the air storage in the concrete tests was lessened in degree, owing to the high relative humidity which existed throughout the storage period.

It should be remembered that the increase in strength resulting from an addition of 5 per cent Cal does not represent the maximum which may be obtained except in very rich mixes. As much as 15 per cent Cal was used in mortar tests, giving an increase of 220 per cent at the 2-day period.

The general effect of Cal on Portland cement mixtures is the same as might be expected from the use of equivalent amounts of hydrated lime and calcium chloride.

The 3-year tests by the Bureau of Standards on concrete gaged with a solution of calcium chloride are sufficient grounds for believing that the addition of Cal will not injuriously affect the ultimate strength and integrity of Portland cement concrete.

WASHINGTON, May 26, 1920.

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POURING AND PRESSURE TESTS OF CONCRETE

BY

W. A. SLATER, Engineer Physicist
Bureau of Standards

A. T. GOLDBECK, Engineer of Tests
Bureau of Public Roads

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POURING AND PRESSURE TESTS OF CONCRETE

By W. A. Slater and A. T. Goldbeck

CONTENTS

	Page
Introduction	3
I. Pouring, consistency, and compressive-strength tests of concrete in cylinders and slabs	3
1. Pouring test	3
2. Strength tests	6
3. Summary	8
II. Measurements of pressure of concrete against forms during pouring	9
1. Introduction	9
2. Description of test	9
3. Results	11
4. Summary	13

INTRODUCTION

This technologic paper presents the results of two minor investigations carried out at the request of the concrete ship section of the Emergency Fleet Corporation. They were undertaken with the object of obtaining data useful in design under the special conditions involved in the construction of concrete ships.

I. POURING, CONSISTENCY, AND COMPRESSIVE-STRENGTH TESTS OF CONCRETE IN CYLINDERS AND SLABS

1. POURING TEST

The object of the pouring test was to determine to what extent a satisfactory job of concreting could be secured under conditions likely to exist in field work during the construction of concrete ships. To be satisfactory there should be no voids in the finished concrete. The work would be expected to have a smooth surface finish, and the concrete should have strength commensurate with the mix and should be of uniform quality. This test was made in the concrete laboratory of the Bureau of Public Roads, Arlington, Va., on May 4, 1918.

The pouring specimen used was a slab 6 feet high, 6 feet wide, and 4 inches thick, having a mass of reinforcement designed to reproduce conditions such as would occur in pouring the shell of a

reinforced concrete ship. Fig. 1 shows the reinforcement assembled and in position with one side of the form temporarily removed.

Wedge-shaped strips cut from 1¼-inch square stock were attached to both of the side forms horizontally at intervals of 12 inches, marking V-shaped grooves along which the slab was later broken into sections for testing transversely. The wedge strips also served the purpose of choking the opening through which the concrete had to pass and therefore made pouring very much more difficult. The percentage of tension reinforcement was taken large enough to insure failure of the concrete in compression in the transverse tests of the slab sections, unless the strength of the concrete, as computed by the parabolic formula, should exceed 3500 pounds per square inch.

An Austin cube mixer having a rated capacity of 1 cubic yard per batch was used for mixing the concrete for this test. The concrete was proportioned by volume, using 1 part of a standard Portland cement, two-thirds part of Potomac River sand, and 1⅓ parts of gravel. The gravel was graded in size from one-fourth to one-half inch. The amount of water used in mixing was 12.5 per cent of the combined weight of the dry materials. The materials were weighed, and the proportions by volume, 1 : ⅔ : 1⅓, were computed from the weights.

Four small batches of concrete were mixed to pour the specimen. Reference to Table 1 shows that there was some variation in the consistency in the different batches. The accidental loss of an unmeasured but small quantity of water during the mixing of the first batch accounts for a stiffness in that batch somewhat greater than that of the succeeding batches.

The cement and aggregate were mixed dry for about 30 seconds after which the water was added and the mixer was run for 1 minute and 45 seconds before dumping the first wheelbarrow load of concrete. It was generally 1 minute more or longer before the remainder of the batch had been removed from the mixer. At the best, an appreciable amount of concrete remained in the mixer from one batch to the next.

The concrete in the lower half of the slab was placed in the form by means of shovels and trowels through an opening in the side of the form 10 inches high and with its lower edge 3 feet 1 inch above the base of the form. The location of this opening is indicated in Fig. 2 by the words "window panel." The form was vigorously pounded with heavy hand hammers throughout the pouring. Air bubbles were released by the pounding and

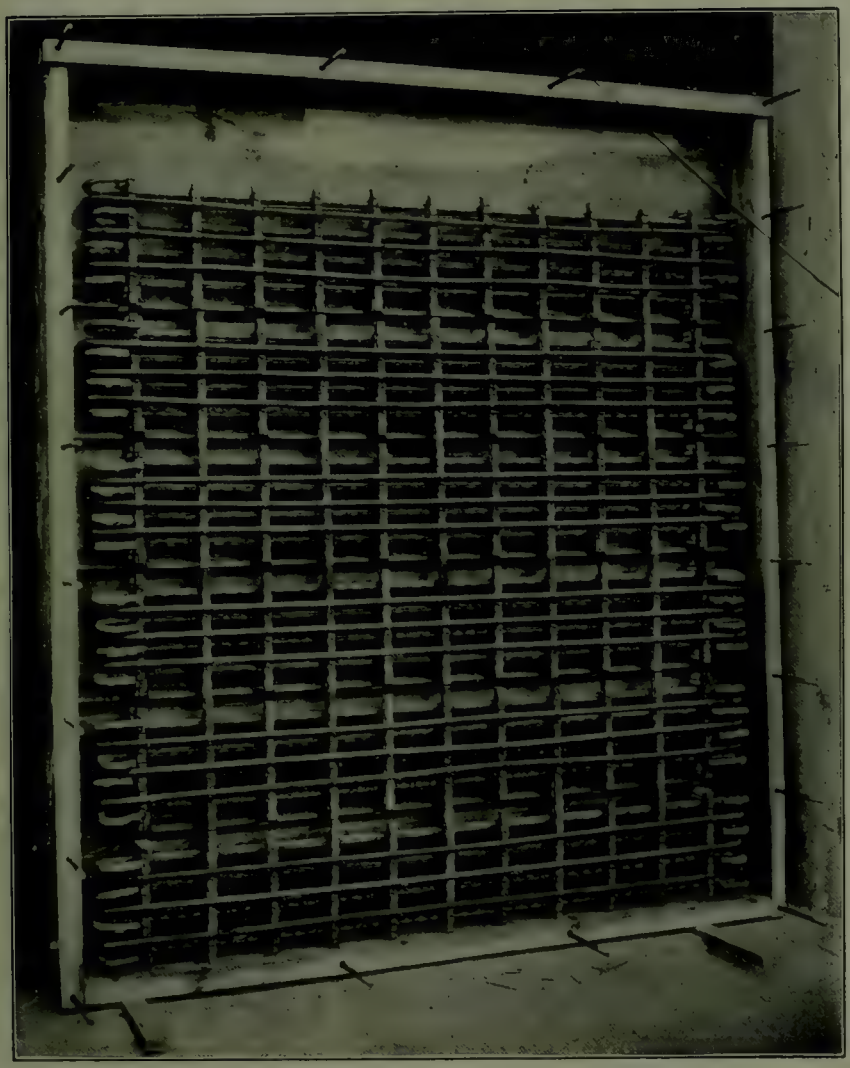

FIG. 1.—*Assembled reinforcement and form for specimen used in pouring test*

FIG. 2.—*Pouring-test specimen removed from form*

rose to the surface of the concrete. The rising of air bubbles continued for a considerable length of time, and had not entirely stopped when pouring was completed and the pounding was discontinued.

During the pouring of the slab an observation was made to find the angle of repose of the concrete. The concrete was poured into the form at one point and allowed to flow toward both ends, assisted to some extent by the pounding outside the form. It was found that the slope of the concrete surface was 30° or more above the horizontal.

In order to check the consistency of the mixture used in the pouring tests, four control cylinders were made. The tests for consistency were made by filling 6 by 12 inch cylindrical molds with concrete taken from the wheelbarrow as it was being wheeled to the form for pouring the slab. The molds were placed on a smooth, clean portion of the concrete floor which had previously been dampened. The molds were then lifted at a uniform rate in a line as nearly vertical as possible. This allowed the plastic concrete to run out into a puddle on the floor. The diameter of the puddle thus formed was taken as a measure of the consistency. After the diameter of the puddle had been measured, the concrete of the control specimen was replaced in the mold and kept for a compression test as a control of the strength of the concrete which went into the slab. The cylinders were removed from the molds after 24 hours and stored under damp burlap until tested. Table 1 gives the consistency readings for the control specimens, and also the compressive strength when tested at the age of 31 days.

TABLE 1.—Consistency and Compressive Strength of Control Cylinders

[Water 12.5 per cent of combined weight of dry materials]

Cylinder No.	Batch No.	Barrow No.	Consistency reading[a]	Maximum load	Compressive strength
			Inches	Pounds	Lbs./in.²
1	(b)	.....	16¼	103 940	3670
2	1	3	10	.....	.....
3	2	2	16	.....	.....
4	2	3	13½	102 570	3620
5	3	1	12¾	.....	.....
6	3	2	11	95 000	3350
7	4	1 and 2	13	114 000	4030

[a] The consistency reading is the diameter of the puddle of concrete remaining after lifting the 6 by 12 inch cylindrical mold.

[b] This specimen was made from a hand-mixed batch just large enough for the specimen, previous to starting the pouring test.

The concrete at the top of the slab was struck off even with the edge of the form in order to provide a means of measuring the amount of settlement during setting. Later observations showed that a settlement of about one-half inch had occurred in a total depth of 6 feet.

One side form was removed the second day after pouring in order to examine the surface of the specimen. This surface is shown in Fig. 2, and its appearance indicates that the pouring was satisfactorily completed. A smooth surface was obtained at all points on the face of the slab. Even under the wedge-shaped strips used to separate the individual slabs only one place was observed where a perfect filling was not obtained. This imperfection was so slight, however, that it is not noticeable in the photograph.

2. STRENGTH TESTS

It was desired to see if any variation existed in the strength of the concrete caused by differences in position in the specimen. Accordingly the specimen was divided into shallow beams for testing separately under transverse loading. A large amount of steel was used for tension reinforcement, with the intention of forcing failure by compression in the concrete. In spite of that provision all of the specimens failed in tension, and the full strength of the concrete was not developed.

Six sections were made by dividing the specimen into beams 12 inches wide and 6 feet long. This separation was made at an age of about 3 days by breaking the slab along the lines of the V-shaped grooves shown in Fig. 2. The beams were numbered from 1 to 6, inclusive, starting with the bottom of the specimen as poured. They were stored under wet burlap and were tested at an age of 31 days.

The reinforcement in each shallow beam consisted of 4 five-eighths inch square corrugated bars in the bottom and 3 one-half inch plain round bars in the top. The top and bottom layers of reinforcement were separated by 11 transverse three-fourths inch square spacing bars 12 inches in length. The amount of bottom reinforcement varied from 4 to 4.7 per cent, on account of the variation in thickness caused by uneven spreading of the form during the pouring. The beams were tested on a span of 5 feet 6 inches, with the load at two points 12 inches apart and each 6 inches from the center of the span. All bearing blocks were bedded in plaster of Paris. Deflections were measured at midspan by means of an Ames dial reading to 0.001 inch.

The measured thickness of the sections into which the slab was divided was greater than the nominal dimension of 4 inches, indicating that there was a slight bulging of the forms due to the pressure of the concrete.

The compressive stress in the concrete at the maximum load, as computed by the parabolic formula, exceeded the assumed value of 3500 pounds per square inch, and none of the shallow beams failed by compression in the concrete. Table 2 gives the results of the beam tests.

TABLE 2.—Results of Shallow Beam Tests

Beam No.	Measured thickness	Depth to tension steel[a]	Tension reinforcement	Maximum load	Deflection at maximum load	Compressive stress at maximum load f_c		Tensile stress at maximum load, straight-line formula f_s	Ratio computed stress at maximum load to working stress	
						Parabolic formula	Straight-line formula		Compression[b] $\frac{f_c}{1500}$	Tension[c] $\frac{f_s}{16\,000}$
	Inches	Inches	Per ct.	Pounds	Inch	Lbs./in.²	Lbs./in.²	Lbs./in.²		
1	4.13	2.75	4.7	10 300	0.99	4460	6320	40 600	4.22	2.54
2	4.60	2.99	4.4	12 580	.91	4770	6630	45 400	4.42	2.84
3	5.88	3.13	4.2	11 630	.68	4080	5650	40 200	3.76	2.51
4	5.19	3.28	4.0	13 290	.58	4240	5950	43 500	3.96	2.72
5	5.13	3.25	4.0	12 430	.86	4250	5650	41 000	3.77	2.56
6	4.44	2.91	4.5	11 280	.91	4520	6250	41 900	4.17	2.62

[a] The depth of tensile steel given assumes that the reinforcement remained as originally placed in the form, and that the form bulged equally in both directions.

[b] In determining the ratio of the compressive stress at maximum load to the working stress the value of f_c used was computed by the straight-line formula, taking n=10 and k=0.6. The working stress was 1500 pounds per square inch. The top layer of bars was not counted as reinforcement as it came practically at the neutral axis.

[c] The ratio of the tensile stress at maximum load to the working stress is the factor of safety against failure by tension in the reinforcement.

The computed compressive stress in the concrete of the shallow beams at the maximum load was greater than the strength of the control cylinders, even though the ultimate strength of the concrete in the shallow beams had not been reached. The average strength of the control cylinders was 3670 pounds per square inch. The average computed compressive stress developed in the beams at the maximum load was 4400 pounds per square inch by the parabolic formula and 6075 pounds per square inch by the straight-line formula. The ratios of the computed flexural stress at maximum load to the cylinder strength were 1.20 and 1.66 for calculations by the parabolic and the straight-line for-

mulas, respectively. These ratios are consistent with results found in previous tests.[1]

Under the caption "Ratio computed stress at maximum load to working stress" the values corresponding to the tensile stresses are the factors of safety of the beams. The values corresponding to the compressive stresses are not factors of safety, because the beams did not fail by compression. It is apparent that the factors of safety against compression failure must have been higher than the ratios under the caption "Compression."

3. SUMMARY

(*a*) The test indicated that with the gravel aggregate used it was practicable to obtain a smooth dense surface, such as is required in the shells of concrete ships, in spite of the narrow and constricted space between forms and the obstruction of the reinforcement.

(*b*) In the pouring test the maximum compressive stress developed in the concrete in flexure gave computed values averaging 66 per cent higher than the ultimate compressive strength of the concrete as determined by tests of control cylinders.

(*c*) The quality of concrete obtained by pouring into forms which were well pounded during the pouring was quite uniform; the maximum loads and computed stresses obtained in flexure tests of shallow beams made from the pouring test specimens were reasonably consistent.

(*d*) No variation in the strength of the beams due to differences in position in the pouring test specimen was determinable on account of the fact that none of the failures occurred by compression.

(*e*) The factors of safety against compression failure are not known, as the beams failed by tension. It is apparent, however, that if sufficient reinforcement had been provided to prevent tension failure, the factors of safety against compression failure would have been found to be greater than the values in Table 2 given as the ratio of the stress computed by the straight-line formula for the maximum load to the working stress.

(*f*) The working stress used in concrete ship design, 1500 pounds per square inch in flexural compression, provides an ample factor of safety against compression failure with concrete of the quailty used in these tests.

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[1] Slater and Zipprodt, Compressive Strength of Concrete in Flexure, Proc. Amer. Concrete Inst.; 1920.

II. MEASUREMENTS OF PRESSURE OF CONCRETE AGAINST FORMS DURING POURING

1. INTRODUCTION

Measurements of the pressure of concrete against forms were made during the pouring of a large reinforced-concrete test specimen. This specimen was poured for the concrete ship section of the Emergency Fleet Corporation at the Bureau of Standards in Washington, D. C., on May 10, 1918. The specimen had a height of 16 feet 11 inches, and at points where the horizontal pressure was measured it had a thickness of 2 inches. The concrete used consisted of 1 part of a standard Portland cement, two-thirds part of Potomac River sand and 1⅓ parts of gravel under one-half inch in size. Proportions were measured by volume. The amount of water used was about 12.5 per cent of the combined weight of the dry materials. The consistency of the concrete was measured in a manner similar to that in the pouring test by lifting a 6 by 12 inch cylindrical mold from a freshly poured control specimen. The measured diameter of the puddle thus formed was taken as a measure of the consistency of the concrete entering into the specimen. The pressure measurements were made by means of pressure cells which have been developed by the Bureau of Public Roads in investigational work on earth pressures.[2]

2. DESCRIPTION OF TEST

Measurements of pressure were taken at seven positions on the specimen during the filling of the form. The locations of the pressure cells are shown in Fig. 3. Cells 1, 2, and 3 were placed on vertical surfaces, and the pressures were measured in a horizontal direction. Cells 4, 5, 6, and 7 were placed on horizontal surfaces and upward vertical pressures were measured. The time was recorded when pouring of concrete began, and during the test a record was made of the elapsed time, the readings on the pressure cells, and the height of the concrete. The height of the concrete was determined by sounding the forms and is, therefore, only approximate. The concrete was hoisted in buckets which were emptied into the form through small windows which were provided for the purpose. These openings were located immediately above the levels of cells 4, 5, 6, and 7. The forms were rapped sharply with hand hammers to settle the

[2] For description of the pressure cells, see Goldbeck, A. T., Proc. Am. Soc. Test. Mats., 17, p. 641, "Distribution of pressures through earth fills." For additional data see Smith, Earl B., Proc. Am. Conc. Inst., 1920, "Pressure of Concrete against Forms."

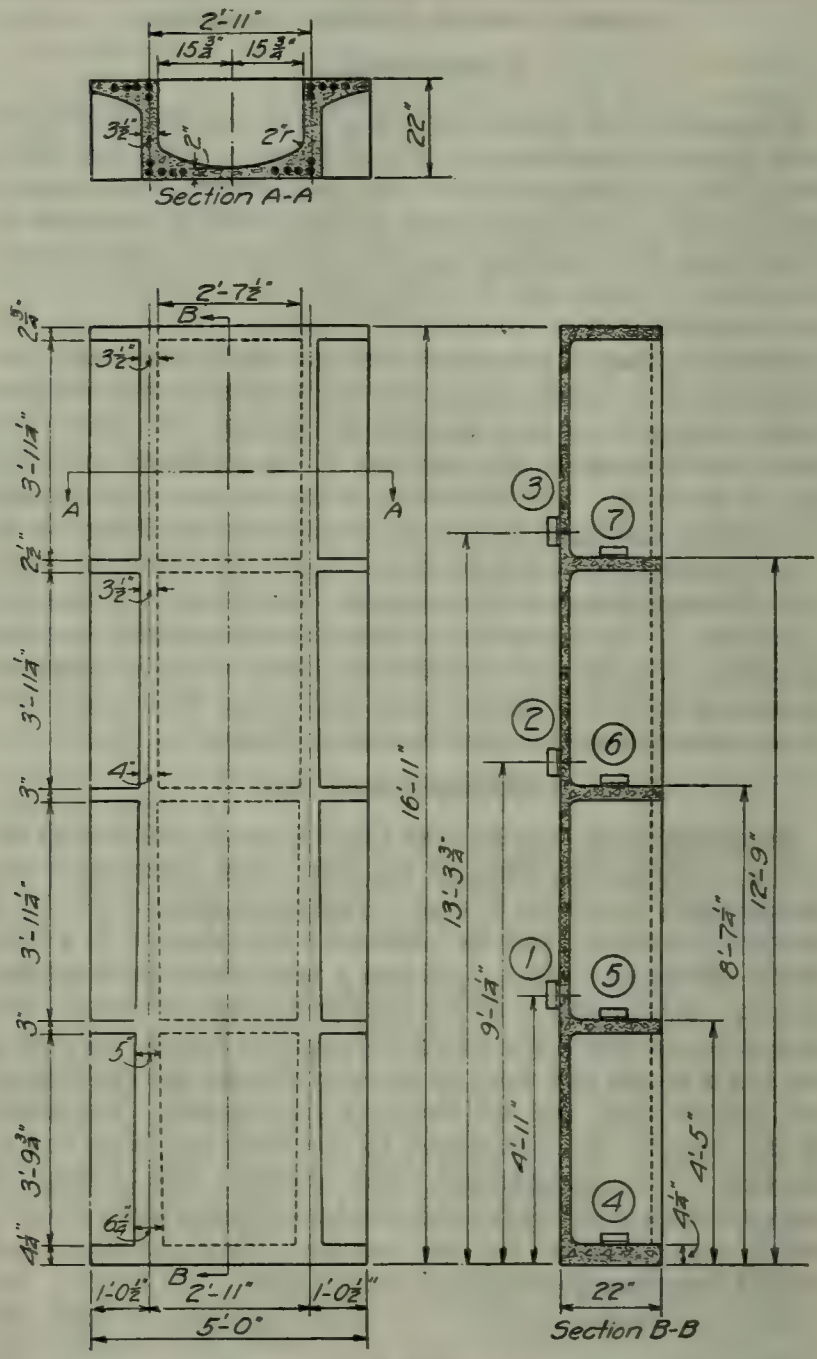

FIG. 3.—*Specimen used in pressure test; location of pressure cells shown by figures within circles*

concrete and to set free the air bubbles. When the forms were removed it was found that a smooth job of concreting had been secured in all except one or two very small areas.

3. RESULTS

The results of this test are shown in Fig. 4. On these diagrams the ordinates represent the head of concrete on the pressure cell and the measured pressure of the concrete against the form. The abscissas represent the time in minutes elapsing after the surface of the concrete was at the level of the pressure cell. In every case the first reading on the cell was taken from 5 to 10 minutes after the concrete had come into contact with the cell.

From the curve shown for cell No. 1, it will be noted that as the head of concrete increased, the pressure also increased almost uniformly up to the maximum observed pressure. However, 30 minutes after the initial reading had been taken, or approximately 35 to 40 minutes after the concrete had reached the level of the cell, the pressure began to decrease in spite of the increasing head of concrete on the cell. Apparently this decrease was because the concrete which was in contact with the cell had begun to harden, causing it to act more in the nature of a solid than of a liquid. Cell No. 1 registered zero pressure about 150 minutes after the initial reading.

The maximum pressure indicated by cell No. 1 was 3.1 pounds per square inch; the head of concrete at that time was about 3.6 feet. Assuming that concrete weighs 150 pounds per cubic foot, and also that it acts as a liquid, its lateral pressure would be 3.6 times 150, or 540 pounds per square foot. As the measured pressure was equivalent only to 446 pounds per square foot, it indicates that the concrete did not act as a perfect liquid. A pressure of 446 pounds per square foot would be produced by a liquid whose weight was only 124 pounds per cubic foot.

In the diagram for cell No. 2 it will be noted that after the first foot of concrete had been poured above the cell level, very little more was placed for about 32 minutes, during which time the original batch had begun to harden and the pressure decreased slightly. After a further interval of 30 minutes an additional 2 feet of concrete was placed in the form, but the pressure on cell No. 2 did not change. Apparently this was due to the fact that the concrete in contact with the cell had already hardened. Zero pressure was registered by this cell at the end of 150 minutes, notwithstanding the gradually increasing head of concrete.

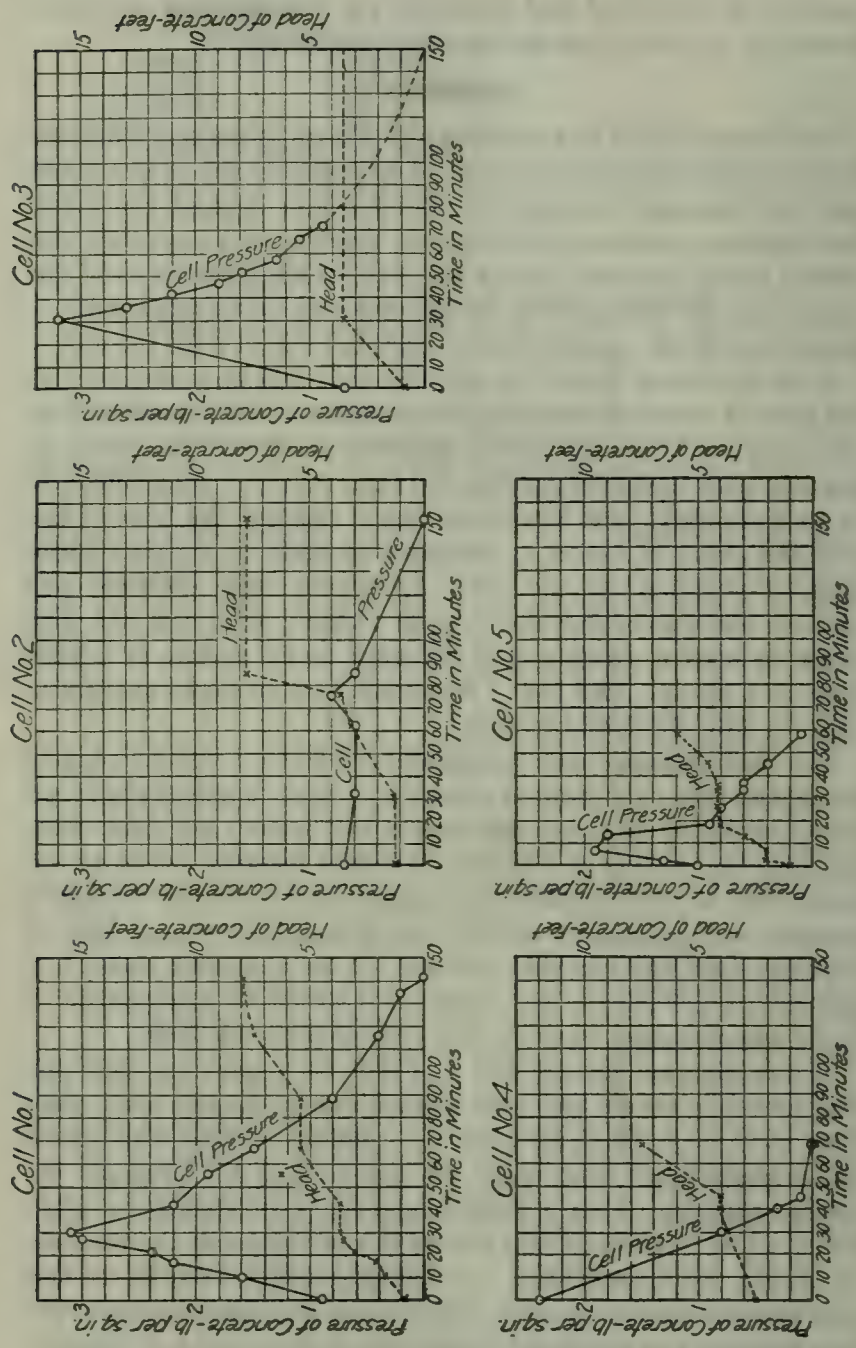

FIG. 4.—*Cell pressure and height of concrete during pouring*

Cell No. 3 registered a maximum pressure of 3.2 pounds per square inch at the end of 30 minutes under a head of 3.6 feet. No more concrete could be added, as the form was full, but additional pressure readings were taken at intervals during the next 60 minutes. Each succeeding reading became lower as the concrete hardened. The curve is shown dotted after the last reading to indicate the probable rate of decrease to zero pressure. Apparently zero pressure would be indicated in about the same length of time as for cells Nos. 1 and 2.

Cells Nos. 4 and 5, which registered upward vertical pressures, present somewhat the same characteristics in the time-pressure curves as the cells measuring lateral pressure, except that they show smaller pressures and reach their maxima sooner. They also registered zero pressure after a shorter time interval.

Only two readings were taken on cell No. 6, and these have not been plotted. Cell No. 7 showed no indication of pressure whatever, and it was found after removal of the forms that no concrete came into contact with the cell.

4. SUMMARY

(*a*) The maximum pressure against the forms during pouring of concrete was found to be equivalent to that of a liquid weighing about 124 pounds per cubic foot.

(*b*) The maximum pressure was found to be that due to the head of concrete existing at the end of about 40 minutes. After that time the pressure gradually decreased in spite of increasing head of concrete.

These tests suggest the desirability of conducting further tests to determine (1) the relation between the pressure on the forms and the rate of increase in the head of the concrete, and (2) the influence of hardening of the concrete upon the pressure under increasing head.

WASHINGTON, June 15, 1920.

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S. W. STRATTON, DIRECTOR

No. 176

SLUSHING OILS

BY

PERCY H. WALKER, Chemist

LAWRENCE L. STEELE, Associate Chemist

Bureau of Standards

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By Percy H. Walker and Lawrence L. Steele

CONTENTS

	Page
I. Introduction	3
II. Nature of slushing oils	4
III. Desired properties of slushing oils	4
IV. Objection to hard and fast requirements as to composition of slushing compounds	5
V. Laboratory tests on slushing compounds	8
VI. Proposed specifications for slushing greases suitable for the protection of exposed bright metal	18
VII. General composition of slushing oils	21

I. INTRODUCTION

Recently the Bureau of Standards has had numerous inquiries regarding information as to methods of protecting from corrosion metal in storage for rather long periods. These requests generally refer to the protection of metal articles which are of such a nature as not to allow the application of metallic coatings, such as galvanizing, or of vitreous enamels, paints, or varnishes. It is obvious that with bearing parts of machinery, coatings such as the above, which form dry and hard films and can not be easily removed, are not suitable. Our attention is therefore confined to a consideration of protective coatings which remain in a soft condition so that they can be easily removed at any time.

Regarding the length of time these coatings will afford adequate protection, there is little information available. It is well known that good oil paint will protect steel for several years when exposed to the weather, and that a good varnish will protect steel for several months. The protection afforded by paint and varnish to articles when kept indoors is very much greater than when exposed to the rays of the sun. While no data based on experiments are available, it is safe to assume that coating with a good grade of varnish and storing protected from the weather may be expected to preserve metal for several years. Paint under similar conditions may be expected to preserve metal for a considerable number of years. As mentioned above, however, these two types

of materials are not applicable for use on the bearing parts of machinery, or on many metallic articles which must be protected from corrosion. Such bright metal is protected by coating it with a layer of nondrying grease, usually known as slushing oil or slushing compound.

II. NATURE OF SLUSHING OILS

Slushing oils or slushing compounds are of a varied nature, sometimes being straight mineral greases (petroleum residuum, such as petrolatum), sometimes mixtures of mineral and animal greases with or without the addition of soap (such as lime rosin or lime fatty acid soaps), finely divided mineral matter, and volatile solvents. Sometimes the petroleum base is mixed with rosin or rosin oils. For example, certain branches of the Army used for a number of years on rifles and guns a compound composed of petrolatum mixed with not over 20 per cent rosin. Mineral oils as fluid as light spindle oils have been used for this purpose, but from the discussion which will follow it will be seen that such thin products are of very doubtful value except for very limited and particular purposes.

From one of the primary requirements of such materials, that they should be easily removed when desired, it follows that it is impossible to get a material of this nature which can not be rubbed off, although there is a very marked difference in ease of removal by abrasion.

III. DESIRED PROPERTIES OF SLUSHING OILS

An ideal slushing compound is one which can be easily applied to all kinds of metal surfaces by a variety of methods. It should coat the surface with a sufficiently thick and impervious film to exclude moisture and air (to prevent rusting), should remain in position for an indefinite length of time and yet be completely removable from the surface without undue labor. The material should itself have no corrosive action on any kind of metal. It is believed that the above statements give the essential requirements of a slushing compound. In addition, however, it is very desirable that the coating formed should be sufficiently transparent so that inspection to detect beginning of corrosion can be made without cleaning the article.

Metal articles which are absolutely protected from moisture, air, and other gases will never corrode. It therefore follows that any coating which will permanently exclude moisture, air, and

any other gases commonly met with in the atmosphere, and which itself has no corrosive action must of necessity protect the metal from rust. There is, of course, no material which will to the fullest extent meet all of the above requirements, but for practical purposes it is believed that a number of materials of the varied composition indicated above answer very well for this purpose. No such materials in very thin films are absolutely impervious to air and moisture, hence very fluid compounds that leave only very thin films on the metal will not give as good protection as the same character of compound of a thicker consistency.

IV. OBJECTION TO HARD AND FAST REQUIREMENTS AS TO COMPOSITION OF SLUSHING COMPOUNDS

It follows from what has been previously said regarding variation in the composition of slushing compounds that any definite limitation on composition of the material desired may exclude from consideration perfectly satisfactory materials which do not meet the particular requirements as to composition and will thus tend to increase price.

In some cases we have noticed composition requirements which not only limit in an unjustifiable manner the choice of material for making a satisfactory article, but in some cases are so worded that it is practically impossible to secure any material which will meet the requirements. For example, we have seen specifications which called for practically pure petroleum products free from even a minute trace of acid. This clause was probably introduced because the acids most commonly thought of, mineral acids and the strong organic acids, are corrosive to metal, and such substances should not be present in any material intended for protecting metal from corrosion. On the other hand, some resin acids have practically no effect on the common metals, and resins containing these acids are frequently very valuable additions to slushing oils, producing products which have superior physical properties to the original petroleum base. Even without the addition of such resins, the petroleum base will never be free from small quantities of acid. It is well known that all petroleum products when exposed to light and air develop more than traces of acid (Waters, B. S. Scientific Papers, No. 153, and Technologic Paper No. 73).

Another requirement which has appeared in certain specifications and which appears to have originated in erroneous application of certain well-known facts is that the material should con-

tain chemicals that are known to be rust inhibitors. It would, undoubtedly, be a good thing to secure a slushing oil to which actual rust-inhibiting properties had been given. Unfortunately, there is no known method of so altering these materials as to render them anything but at best inert in themselves. Regarding the inhibition of rust, it is of course well known that certain chemicals under particular conditions produce a so-called passive condition of iron, leaving the surface in a condition which resists rust for limited periods of time or resists rust under particular conditions of exposure. The well-known example of treating iron with very concentrated nitric acid might be mentioned, and of course it is ridiculous to consider that nitric acid, because under certain conditions it renders iron passive, would be a good material to add to a material for protecting iron from rust.

Iron and steel immersed in water solutions of strong alkalies will not rust as rapidly as when immersed in pure water. The water solutions of soluble chromates are among the best known rust-inhibiting substances, and if iron or steel is kept immersed in an aqueous solution of soluble chromate, it will resist rust for a much longer time than the iron immersed in water. This property of soluble chromates appears to have been the basis of the idea that rust-resisting chemicals could be satisfactorily added to slushing oils. One can not predict the action of chemicals when incorporated in any way with an oily medium from the action of these same chemicals when in water solution, as is shown very clearly by some very interesting experiments which were conducted on paints.

It had been found that some paint pigments, when suspended in water, had a marked influence on the speed with which iron and steel immersed in the water rusted. A large number of paint pigments were tested in this manner, and it was found that they could be divided into three classes—those in which the iron or steel rusted very much slower than in pure water, which for sake of convenience were called water inhibitors; those in which the iron or steel immersed in water containing pigment rusted in about the same degree as in pure water, which were called indeterminates; and those in which the iron or steel immersed in water containing the pigment rusted distinctly more rapidly than when in pure water, which were called water stimulators. This fact attracted so much attention that very extensive tests were made to ascertain whether these same pigments in an oily vehicle would behave in the same manner as they did in suspension in water. These

tests were carried out under the direction of two committees of the American Society for Testing Materials.[1] The committees of this society carefully inspected these tests, which extended over a period of more than five years, and in rendering their final report made the following statement:

> The summarized results indicate that the water test as at present developed can not be depended upon to give accurate information as to the protection which pigments will or will not afford metal structures when such pigments are applied in oil media. The summarized results also indicate that the arbitrary designations, inhibitive, indeterminate, and stimulative, as referring to the results of the water test, are not always applicable to the same pigments when used with linseed oil for protective coatings.

A single illustration taken from tests on numerous pigments by the above-mentioned committee will convince one that this conclusion was entirely justified as respects paints. Among the pigments selected were two samples of Prussian blue, Nos. 44 and 45. No. 44 Prussian blue was intentionally selected as one which in the preliminary water test acted as a stimulator of corrosion. No. 45 was one which under the water test acted as an inhibitor of corrosion. This Prussian blue No. 45 contained some chromate. In June, 1913, before the conclusion of this test, it was necessary to remove some of the panels, and the committee selected for discontinuing a number of panels which at that time were considered as having failed; among them was the inhibitive Prussian blue No. 45, whereas the stimulative Prussian blue No. 44 was not included in this list. It is probable that these observations were never carefully considered by the people who have advocated the use of so-called rust-inhibiting chemicals in slushing oils. So far as the writers know, all manufacturers who have attempted to produce slushing oils containing rust-inhibitive chemicals have prepared a product which was essentially an emulsion of a water solution of sodium or potassium chromate in a straight petroleum residue base. A careful consideration of the nature of this mixture would lead one to the conclusion that the water solution of the chromate could not possibly have any beneficial effect. Such a material when completed would consist of a large quantity of heavy petroleum product with minute globules of water solution of chromate distributed through it. These globules do not come in contact with the metal on which the slushing compound is coated and hence can have no effect as rust inhibitors. Soluble chromates

[1] A. S. T. M. Proceedings, **IX**, p. 106, 1909; **X**, p. 75, 1910; **XI**, p. 192, 1911; **XIII**, p. 369, 1913; **XIV**, Part 1, p. 259, 1914; **XV**, Part 1, p. 222, 1915.

frequently cause serious injury to the skin, and some users of slushing oils containing chromates have been obliged to abandon them on this account.

V. LABORATORY TESTS ON SLUSHING COMPOUNDS

A few months after the United States entered the Great War, the Bureau of Standards started to receive samples of rust preventives from branches of the War Department. This material was intended for use on guns, rifles, and small arms and was purchased on a somewhat indefinite specification. At the request of the War Department the following tests were made on these samples: (1) Test for mineral acid; (2) test for rosin; and (3) an adhesion test.

A test was made for mineral acid by heating some of the sample with distilled water and qualitatively testing the water portion for acid in the usual way. Rosin was detected by the Liebermann-Storch reaction. The adhesion test was performed as follows: The sample was smeared on both sides of a tared copper plate 2 inches square and 0.0063 inch in thickness. This plate was then suspended by one corner in an oven and maintained at a temperature of 65° C (149° F) for 30 minutes. After cooling, the plate was weighed. An increase in weight of the plate of not less than 65 and not more than 85 mg was required.

The above tests probably gave very little information as to the merits of a given slushing oil. From results of work done later, it is believed that no adhesion test, where the film of oil remaining adherent to a test plate under set conditions is weighed, can be relied upon to differentiate between good and bad rust preventives.

Practically all the samples of slushing oil examined for the War Department by the methods outlined above were straight mineral oils of the consistency of petrolatum and did not contain thinners or mineral matter.

During the summer of 1918 the Bureau of Standards received at different times various samples of rust preventives from the War Department with a request in each case that certain extravagant rust-inhibiting qualities claimed by the manufacturer be investigated.

These compounds were analyzed in so far as it was possible and in addition it was thought best that some comparative test be made, using one of the regular samples of rust preventive in use by the War Department as a standard of comparison. To

this end exposure tests on bright steel plates were made. These exposure tests have proved to be of very great value in the evaluation of rust preventives, and the details of the method will be explained at this point: Bright cold-rolled steel of No. 26 gage was cut into pieces measuring 4 by 6 inches and carefully cleaned by the use of cotton waste and benzene. The sample of rust preventive was then applied to these plates either by dipping, brushing, or smearing on with a spatula, according to the nature of the material. It was found convenient to tack the plate to a board before applying the slushing oil, so that the coated plate could be carried without danger of disturbing the film of rust preventive. The coated plates were allowed to stand for one day in the laboratory in an upright position and were then exposed outdoors at an angle of 45°, facing south. They were inspected at intervals of every few days and a record kept of the condition of the various plates.

These exposure tests proved to be of great value in the comparison of a number of samples. The length of time before rust appeared on the plate was influenced very largely by the kind of weather and the season of the year, so that the tests were only comparative. These first exposure tests indicated one fact which has been confirmed in nearly all cases by later series of tests, namely, oils of thin consistency give very poor protection to steel exposed to weather as compared with oils of greater body and hence greater adhesiveness. This is a conclusion which seems most logical.

At the time when these first exposure tests were made, an interesting phenomenon was noted. A compound was being examined, the manufacturers of which claimed it possessed rust-inhibiting qualities. This material was found to be essentially an emulsion of a small amount of a water solution of a chromate in heavy mineral oil The rust-inhibiting properties of chromates in slushing oils have already been discussed on page 6. When this sample was applied in the usual way to bright steel plates, either by smearing on the compound from the original container or else by applying the melted material, and the plate allowed to stand in an upright position in the laboratory, it was noted that the film of rust preventive slipped down the surface of the metal. The material did not melt on the panel and run off, but the whole film moved down the plate like a glacier and left the surface of the metal bare. At first it was believed that the slipping of this

particular rust preventive was due to the small amount of water present in the oil chromate solution emulsion. However, other slushing oils were found later which were free from water and which showed the same phenomenon of slipping from bright metal surfaces.

The cause for this slipping observed in cases of certain rust preventives has not been determined. It is readily seen that a property of slipping from bright metal is a very serious defect in a slushing oil. At the suggestion of the Bureau of Standards, certain Government specifications have included the following clause or a similar requirement:

When a highly polished steel plate 3 by 5 inches, coated with rust preventive, is allowed to stand on the shortest side, with the longest side vertical, for one week, there shall be no slipping of the rust preventive and no portion of the steel surface shall be exposed through movement of the protective coating.

The above clause, it was believed, would eliminate those rust preventives which possessed the serious defect of slipping.

After the signing of the armistice different branches of the War Department became concerned in the problem of storage of guns, airplane engines, machines, etc. The Bureau of Standards received numerous requests for information as to the best methods for the protection of metal from corrosion during storage for long periods of time. Mimeographed sheets entitled "Notes on Protection of Metal in Storage" were prepared and issued on January 4, 1919. In these notes tentative specifications were outlined for three different types of rust preventive: Brushing consistency, dipping consistency, and an opaque compound for special uses.

These specifications did not dwell upon composition of material, but an attempt was made to include certain laboratory tests which, in conjunction with the comparative exposure tests, should give a basis for the evaluation of different samples of slushing oils.

A new adhesion test was prescribed which differed considerably from the War Department method previously described on page 8. This modified adhesion test was as follows:

(*a*) Two polished copper plates 5 by 5 cm and not more than 1.0 mm thick, will be weighed and then coated on both sides with the slushing oil. One of these plates will be suspended by one corner for 30 minutes, in an oven at 75° C, the other in an oven at 65° C. The two plates will then be weighed. The oil remaining on the plate heated at 65° C shall weigh not less than 60 mg and the oil remaining on the plate heated to 75° C shall not be less than three-fourths as much as that remaining on the plate heated to 65° C.

(*b*) When applied to a polished metal plate 10 by 15 cm and the plate then held in a vertical position at any temperature between 15° and 35° C for 5 days, the coat of slushing oil shall remain adherent to the metal, shall not slide appreciably from the top edge of the plate nor leave any portion of the surface.

The clause which required that 75 per cent as much material remain on the plate at 75° C as remained at 65° C was designed to eliminate those rust preventives which rapidly lose their adhesive qualities with a slight increase in temperature. The weight of material required to remain on the plate after 30 minutes at 65° C was arbitrarily taken at 60 mg or more in the case of slushing oils of brushing consistency and 30 mg or more in the case of slushing oils of dipping consistency.

Experience with these adhesion tests has shown them to be of doubtful value in the examination of slushing oils. This point will be discussed in full further on.

In the question of acid content of rust preventives a very radical departure was made. Most slushing oil specifications required that "no acid be present." As already pointed out on page 5, pure mineral oils are likely to develop traces of acid on exposure to light and air. Furthermore, the detection of traces of acid in a petroleum is not certain by present methods. For these reasons it was not specified that no acid be present, but a requirement was added in the specification that "the rust preventive in question should not corrode bright copper, brass, nickel, or iron after five days." It is firmly believed that this requirement in regard to corrosion effectively excludes any slushing oils which contain harmful acid in quantity sufficient to cause trouble in actual use. Harmful acids which may be present in rust preventives cause stains to appear upon bright copper and brass in a comparatively short time, and therefore these metals act as sensitive indicators of the presence of corrosive substances in the slushing oil.

The exposure tests made on bright steel and already described in some detail are believed to be the most important of all the tests proposed in the new tentative specifications for slushing oil.

Since almost any rust preventive in use may be subject to exposure to the weather, the most vital property of a good slushing oil should be that it give an impervious and lasting film on metal, even when subjected to the action of rain, snow, and rapidly changing temperature. The great objection to exposure tests is the length of time that is needed to make them properly. Plates must be exposed for at least 30 days, and better for a space

of two months, in order to draw any valuable conclusions. The time of the year must also be taken into account, because hot summer weather with rain is much more severe on rust preventives than cold winter weather with snow and ice.

A few months after the circular "Notes on Protection of Metal in Storage" had been issued, one of the purchasing bureaus of the War Department sent in a large number of samples of slushing oil with a request that an examination of these materials be made to determine if possible which brands were best suited for rust prevention. There were 44 samples in all, and tests were begun at once, using the methods of test outlined in the specifications of the circular of January, 1919.

The 44 samples were first grouped into three classes: Class 1, the transparent materials of brushing consistency at temperatures above 10° C (50° F); class 2, the opaque materials of brushing consistency at temperatures above 10° C (50° F); class 3, the materials of dipping consistency at temperatures above 10° C (50° F).

Each one of the 44 samples was examined under all the laboratory tests given in the circular, and in addition each material was ashed and the percentage and nature of the ash determined, the acid number was determined, and a qualitative test for rosin was made in each case.

The results of the laboratory tests will be found in Table 1.

Exposure tests lasting for a period of 60 days were made on all the samples. The materials which flowed readily at room temperature were applied by flowing an excess of oil over the plate and allowing the surplus to run off. The materials which were semisolid at room temperature were applied in two ways: (1) One set of plates was coated by smearing on an excess of material as it came from the container; (2) another set of plates was coated with melted material. The plates were coated on July 18, 1919, and exposed to the weather on July 19, 1919. The weather conditions during the following 60 days were unusually severe. There were many hard rains with intervals of hot sunny days. Some of the materials which did not stand up well in the exposure tests might give good protection under less severe weather conditions, but the tests were intended to be severe. It is believed that a satisfactory rust preventive should stand up well on metal for at

least 60 days when exposed to the most severe weather conditions which may prevail at points where the material is to be put in service.

The results of the exposure tests upon the 44 samples after 60 days on the roof are given in Table 2.

In order to compare the results obtained in the exposure tests with the results in the laboratory tests, Table 3 was prepared. It must be kept in mind that several of the rust preventives of semisolid consistency were applied to the steel plates in the exposure test both as a paste and in a melted condition. Where different protection was given, depending upon the mode of application, that sample of slushing oil has been counted twice and the same compound may appear, for example, listed as both "fair" and "failure."

It is a fact worthy of note that nearly all the compounds of semisolid consistency gave the best protection in the exposure test when they were applied to the steel plate in a melted condition.

An examination of Table 3 shows that a strict interpretation of the laboratory tests given in the tentative specification of January, 1919, would have excluded many compounds which showed up well on exposure and would have passed at least three compounds which failed in the exposure test. For example, if the five compounds excluded in the laboratory tests because the resulting film was not removable with kerosene are not considered, we find that a strict interpretation of the adhesion and slipping tests alone would have excluded one compound rated as perfect in the exposure test, one compound rated as good, and eight compounds rated as fair, while two samples rated as complete failure and one sample rated as partial failure would have been accepted from the results of the laboratory tests alone.

The above comparison shows clearly that it was unwise to depend entirely upon laboratory tests outlined in the tentative specifications for decision in regard to acceptance or rejection of rust preventives.

TABLE 1.—Laboratory Tests on 44 Samples of Slushing Oils

B. S. No.	Slipping	Corrosion			Adhesion			Ash		Acid No.	Rosin	Film
		Steel	Copper	Brass	At 65° C	At 75° C	Per cent	Per cent	Contains			
					Mg	Mg						
55784	O. K.	O. K.	O. K.	O. K.	57.6	34.2	59.5	0.07	Fe	27.7	Not detected	
55785	Slipped slightly on steel; did not leave surface.	do	do	do	91.0	71.2	78.4	.07	do	.12	do	Black opaque.
55786	O. K.	do	Corroded	Corroded	50.1	46.3	92.4	.06	do	41.9	Present	
55787	Slipped badly	do	O. K.	O. K.	83.3	64.3	77.2	.54	Na, Cr	.19	Not detected	
55788	do	do	do	do	60.1	59.7	99.4	.34	do	.19	do	
55789	do	do	do	do	83.6	45.4	54.2	.70	do	.12	do	
55790	O. K.	do	do	do	50.3	31.1	61.8	.00		(a)	do	
55791	Slipped badly	do	do	do	71.8	52.4	73.0	.01		.06	do	
55792	do	do	do	do	105.3	64.3	61.0	.67	Ca	.12	do	
55793	Slipped	do	do	do	76.2	39.6	52.0	.04		.25	do	
55794	O. K.	do	do	do	268.0	264.4	98.6	.22		.37	do	
55795	do	do	do	do	28.0	27.6	98.5	.00		.12	do	
55796	do	do	Slightly corroded	Slightly corroded	134.4	97.0	72.0	.20		.19	do	Opaque.
55797	do	Rusted under film	O. K.	O. K.	32.2	27.2	84.5	.00		.03	do	
55798	do	O. K.	do	do	678.6	668.6	98.5	.50		1.99	do	Black opaque.
55799	do	do	do	Corroded	30.4	30.0	98.7	.50	Mn, Ca	7.2	Present	
55800	do	do	do	O. K.	146.2	110.8	75.6	.60	do	5.4	do	
55801	do	do	do	do	2,025.0	1,979.0	97.6	2.85	Fe, Ca	.31	Not detected	Do.
55802	do	do	do	do	106.2	85.2	80.4	24.0	Ba, Zn, S, SO_4.	.19	do	White opaque.
55803	do	do	do	do	101.0	83.2	82.3	7.00	Pb	.25	do	Opaque.
55804	Slipped	do	do	do	46.8	46.0	98.3	.03		.12	do	
55805	O. K.	do	do	do	348.2	294.2	84.5	18.00	Pb, Fe	.68	do	Dark brown opaque.
55806	do	do	do	do	38.0	37.8	99.5	.24	Mn, Pb, Ca	13.6	Present	
55807	Slipped badly	do	do	do	84.0	65.8	78.3	.00		.12	Not detected	
55808	O. K.	do	do	do	89.4	71.4	79.8	.00		.19	do	
55809	do	do	do	do	67.0	48.0	71.6	.00		.03	do	
55810	Slipped badly	do	do	do	44.4	43.4	97.7	.01		.03	do	
55811	O. K.	do	do	do	47.2	47.0	99.5	.00		.03	do	
55812	do	do	do	do	52.8	51.8	98.2	.00		.37	do	
55813	do	do	Corroded badly	do	45.4	45.2	99.5	.00		22.60	do	

55814	....do....	....do....	O. K.	....do....	50.8	49.2	96.8	.06	....	3.50	Present.
55815	....do....	....do....	....do....	....do....	56.2	37.4	66.5	.....	....	.....	Not detected.
55816	Slipped badly.	....do....	....do....	....do....	78.8	57.8	73.4	.00	....	.03	....do....
55817	Slipped slightly.	....do....	....do....	....do....	70.4	70.4	100.0	.00	....	.00	....do....
55818	Slipped badly.	....do....	....do....	....do....	75.5	37.0	75.5	.00	....	(a)	....do....
55819	O. K.	....do....	....do....	....do....	47.6	48.2	101.3	1.30	Mn,Pb,Ca.	9.27	Present.
55820	....do....	....do....	....do....	....do....	138.2	135.2	97.8	.00	....	16.2	....do....
55821	....do....	Rusted under film.	....do....	....do....	36.6	35.4	96.8	.00	....	.03	Not detected.
55822	....do....	Rusted slightly under film.	Slightly corroded.	....do....	40.0	38.2	95.5	.00	....	.03	....do....
55823	....do....	....do....	....do....	Corroded.	121.0	85.5	70.6	.00	....	12.6	....do....
55824	....do....	O. K.	Corroded badly.	Corroded badly.	181.2	144.4	79.7	.00	....	18.0	....do....
55825	....do....	....do....	....do....	....do....	126.8	87.8	69.4	.00	....	16.4	....do....
55826	....do....	....do....	....do....	....do....	125.0	104.8	83.8	.01	....	22.1	....do....
55827	....do....	....do....	O. K.	O. K.	118.2	81.6	69.0	.06	....	.50	....do....

55814	
55815	
55816	
55817	
55818	
55819	Set to touch.
55820	Do.
55821	
55822	
55823	
55824	
55825	
55826	
55827	Opaque.

a Slightly alkaline.

TABLE 2.—Results of Exposure Tests for 60 Days upon 44 Samples of Rust Preventive Received from the War Department on July 11, 1919

B. S. laboratory No.	Materials [a]	Appearance of first rust	Condition of plate after 60 days [b]
55784	L	After 2 days	Complete failure.
55785	L	After 35 days	Partial failure.
55786	L	After 2 days	Complete failure.
55787	S	do	Fair.
55787	M	After 22 days	Do.
55788	L	After 4 days	Complete failure.
55789	S	After 2 days	Partial failure.
55789	M	After 4 days	Fair.
55790	L	After 2 days	Complete failure.
55791	S	After 3 days	Partial failure.
55791	M	After 60 days	Good.
55792	S	After 46 days	Fair.
55792	M	After 27 days	Partial failure.
55793	S	After 46 days	Fair.
55793	M	After 26 days	Do.
55794	L	After 60 days	Good.[c]
55795	L	After 2 days	Complete failure.
55796	L	do	Do.
55797	L	do	Do.
55798	L	None	Perfect.[c]
55799	L	After 4 days	Complete failure.
55800	L	After 60 days	Good.
55801	S	None	Perfect.
55801	M	After 32 days	Fair.
55802	S	None	Perfect.
55802	M	do	Do.
55803	S	After 2 days	Partial failure.
55803	M	do	Fair.
55804	S	do	Partial failure.
55804	M	do	Complete failure.
55805	S	None	Perfect.
55805	M	do	Do.
55806	L	After 18 days	Complete failure.
55807	S	After 2 days	Partial failure.
55807	M	After 46 days	Fair.
55808	L	After 10 days	Complete failure.
55809	S	After 14 days	Partial failure.
55809	M	After 2 days	Do.
55810	S	do	Do.
55810	M	do	Do.
55811	S	do	Fair.
55811	M	After 18 days	Do.
55812	L	After 2 days	Complete failure.
55813	L	do	Do.
55814	L	None	Perfect.
55815	S	After 2 days	Fair.
55815	M	do	Do.
55816	S	do	Complete failure.
55816	M	do	Do.
55817	S	After 10 days	Fair.

[a] Materials marked "L" were liquids at room temperature and were applied to the plates as such; materials marked "S" were semisolids and were smeared on the plates as a paste; in addition the semisolid materials were melted and applied to plates as liquids, and in such cases the mark "M" appears.

[b] Ratings are given as follows: "Perfect," where the exposed steel plate shows no evidence of rust; "good," where the plate shows evidence of only a superficial and slight amount of rust; "fair," where the plate shows decided evidence of rust, but where a large percentage of the surface is still free from rust; "partial failure," where at least one-third of the surface of the plate is covered with rust; "complete failure," where the entire surface of the plate is covered with rust.

[c] At the end of the 60 days' exposure this coating could not be readily removed by waste wet with kerosene.

TABLE 2.—Results of Exposure Tests for 60 Days upon 44 Samples of Rust Preventive Received from the War Department on July 11, 1919—Continued

B. S. laboratory No.	Materials	Appearance of first rust	Condition of plate after 60 days
55817	M	After 14 days	Fair.
55818	S	After 2 days	Partial failure.
55819	M	....do	Do.
55820	L	After 3 days	Complete failure.
55821	L	None	Perfect.[a]
55822	L	After 2 days	Complete failure.
55823	L	....do	Do.
55824	L	After 22 days	Fair.[a]
55825	L	After 27 days	Do.[a]
55826	L	After 22 days	Do.[a]
55827	L	After 2 days	Complete failure.
		....do	Do.

[a] At the end of the 60 days' exposure this coating could not be readily removed by waste wet with kerosene.

TABLE 3.—Relation Between Ratings of Exposure and Results of Laboratory Tests upon 44 Samples of Rust Preventive from the War Department

Rating in exposure	Compounds	Passed all laboratory tests	Failed adhesion test	Failed slipping test	Failed corrosion test	Failed both adhesion and slipping tests	Failed both adhesion and corrosion tests	Film not removable with kerosene
Perfect	6	3	1	0	0	0	0	2
Good	3	1	0	0	0	1	0	1
Fair	13	2	3	3	1	3	2	0
Partial failure	10	1	1	3	0	5	0	0
Complete failure	19	2	4	1	5	2	3	2

Exposure tests on steel panels seemed to be a satisfactory method for distinguishing good slushing oils from poor ones, but this method was open to at least two very serious objections. In many cases the decision as to acceptance or rejection of rust preventives must be made in less than 60 days, and in some cases it would not be possible to await the results of an exposure test for a period of even 30 days. The results of exposure tests on rust preventives are influenced to a very great extent by weather conditions. Hot summer weather is a much more severe test upon a slushing oil than cold winter weather, and it is conceivable that a compound might be accepted upon the basis of 60 days exposure in winter, while the same material might be rejected from the results of summer exposure.

It was therefore seen to be very important that some laboratory tests be devised which would tell in a short time whether a given rust preventive would protect exposed metal under any conditions of weather, especially the severe conditions met with

during the summer near the seacoast with the accompanying salt-laden air. In some of the work already described it had been noticed that certain compounds seemed to be literally washed from the exposed steel plate by the first heavy rainfall. To distinguish this fatal defect in slushing oils, an artificial rain test was devised which will be described in detail further on.

It was considered that the failure of rust preventives which were not washed off the plate by rainfall was due to some one or more of three things: Either the material itself corroded metal, or it was more or less porous and allowed moisture to penetrate the film, or the rust preventive did not remain adherent to the metal owing to slippage or melting off of the coating by the heat of the sun.

It was believed that the detection of corrosive materials in the rust preventive itself was covered in an efficient manner by the corrosion test given in the original tentative specifications of January, 1919, and no change was made in this test.

A so-called oven salt-spray test was devised in which an attempt was made to simulate to a certain extent the combined conditions of hot weather and salt-laden air near the ocean. This new test was also designed to show up rust preventives which were porous or possessed the defect of slipping.

A specification for slushing oils was drawn up which embodied the new oven salt-spray test and the rain test. A comprehensive examination of a series of rust preventives was made according to these tests, and the specification was altered in certain details until the form given below was the final result.

VI. PROPOSED SPECIFICATIONS FOR SLUSHING GREASES SUITABLE FOR THE PROTECTION OF EXPOSED BRIGHT METAL

1. The material shall furnish a coating that shall firmly adhere to all metal surfaces at all temperatures at which they may be exposed under natural conditions.

2. The coating shall permanently remain in such a condition that it can be readily removed with cotton waste wet with kerosene.

3. CORROSION TEST.—When applied to polished iron, steel, brass, and copper surfaces and exposed at any temperature below 100° C (212° F) for a period of not less than five days, there shall be no stains on the metal or other evidence of corrosion due to the slushing oil.

4. OVEN SALT-SPRAY RAIN TEST.—Four bright steel plates approximately 4 by 6 inches shall be tacked to separate boards and thoroughly cleaned by the use of benzene. The slushing oil to be examined shall be melted, if not already liquid, and an excess flowed over the plates. One of the plates shall be kept in a vertical position in the laboratory for 24 hours and then placed in a rack on the roof so that the plate shall be inclined at an angle of 45° to the vertical, facing south. The plate shall be sprayed lightly with a 3 per cent salt solution on the first day. The other 3 plates shall be hung in a vertical position in an oven and maintained at a temperature of 45 to 50° C (113 to 122° F) for at least 48 hours. The plates shall be removed from the oven, allowed to cool, and 1 plate exposed on the roof in the rack described above. Another plate shall be kept in a horizontal position and lightly sprayed with a 3 per cent salt solution once every day for a period of 5 days. The fourth plate shall be placed under an intermittent shower, a vigorous shower being applied for approximately 3 to 4 minutes, then no water for about the same length of time. The shower is to be formed by allowing water to siphon at intervals from a 5-gallon tank into a metal trough, the bottom of which has three or four parallel rows of small holes (about one-sixteenth inch in diameter). The water should fall about 2 feet from the trough to the plate. This test should be continued for not less than 5 hours, the plate being held in a position about 60° to the vertical immediately under the falling water. At the end of 5 hours of this intermittent showering, the plate shall be placed in a horizontal position and allowed to remain with any adhering water for at least 24 hours. No rust shall be in evidence on any of the 4 plates after 5 days.

When time permits, the plates on the roof should be exposed for a period of 60 days, and at the end of this time there should be no appreciable rust in evidence.

DISCUSSION.—It is believed that any material which passes all the tests and requirements given above will protect bright exposed metal for a period of at least 60 days in any weather commonly encountered and will probably continue to protect exposed metal for many months. It has been found that the compounds which pass the above tests are usually greases of rather high melting point and should be applied to metal while melted, either by spraying or by dipping. We have seen a few samples that are liquid at ordinary temperatures which fulfill the requirements.

In order to gain some insight into the way in which the new laboratory tests on slushing oils would work out in routine examination of rust preventives, tests were made upon 23 of the 44 samples from the War Department previously referred to. Those compounds which had been found to corrode metal in the original tests were not selected. Compounds which had been found to give a varnish-like film were also left out. The results of exposure of each one of these 23 compounds during the most severe weather in Washington (July and August) was known. Each of these 23 compounds was carefully put through the oven salt-spray rain test outlined in the new proposed specification.

Seven of the 23 samples had been rated as "complete failure" in the 60-day exposure test. All 7 of these slushing oils failed conclusively in the oven salt-spray rain test. Seven of the 23 samples had been rated as "partial failure" in the exposure test. Six of these samples failed conclusively and 1 passed the oven salt-spray rain test. Four of the 23 samples had been given a rating of "fair" in the exposure test. All of these samples passed the oven salt-spray rain test. One of the 23 samples had been rated as "good" in the exposure test and it passed the oven salt-spray rain test. Four of the 23 samples had been rated as "perfect" in the exposure test. Three of these samples passed the oven salt-spray rain test, while the other sample showed slight evidence of rust on the edge of the plate.

From the above results it is seen that the laboratory tests in the new proposed specifications would exclude 13 out of 14 samples which had been rated as unsatisfactory in the exposure test. At the same time 1 sample out of 9 of the compounds rated as satisfactory in the exposure test would be excluded.

The results obtained above on the 23 samples by the laboratory tests in the new proposed specification are believed to average as well as most laboratory tests when they are compared with the results of actual service conditions or conditions similar to actual service.

Laboratory tests are almost never perfect, but it is believed that the tests outlined in the proposed specification are as good as any that can be given at the present time. Attention is called to the fact that even where it is possible to run exposure tests extending over considerable periods of time and some brand of material is found to give good service, rapid laboratory tests are still of great use in determining uniformity of quality of the product. It has often been noticed that different lots of the same

brand of rust preventive received at different times show wide variation in rust-preventing qualities. A rapid laboratory test can be used to determine the quality and uniformity of each shipment of slushing oil.

VII. GENERAL COMPOSITION OF SLUSHING OILS

During this investigation comparatively little work was done upon the composition of slushing oils. It was felt that it would be unwise to attempt in any specification for these materials to limit the manufacturer in the raw materials that he should use, because performance tests were more important than composition requirements.

Only a brief discussion of the general composition of commercial slushing oils will be given here, together with a description of some experimental batches of slushing oil prepared during the course of this investigation.

An examination of Table 1, on page 14, where the results of chemical tests upon 44 commercial samples of rust preventive are given, will give considerable insight into the composition of these samples. The majority of these materials showed a low acid number, together with a low ash content. These compounds are probably straight petroleum products of varying consistency from thin oils of low viscosity to petrolatum-like materials and thick petroleum residues.

The compounds which yield a considerable amount of ash may contain mineral matter as a filler (for example, one slushing oil seemed to be a mixture of lithopone and a heavy petroleum residue, while another seemed to be mainly iron oxide with a petroleum product), or they may be petrolatum-like compounds emulsified with a little water soluble chromate solution, supposed to act as a rust inhibitor.

The presence of small amounts of manganese with or without lead and calcium would indicate that some of the compounds are varnishes or mixtures of a petroleum product with varnish.

Several of the materials with high acid number were found to be blown vegetable oils. There were several black opaque compounds, which consisted of soft asphalt material or hard asphalt thinned with some solvent.

Rosin was detected in several of the compounds. In some cases it was present as an ingredient of varnish, while in other cases the slushing oil seemed to be a mixture of a petroleum product with rosin.

Among the 44 samples there was one, Bureau of Standards laboratory No. 55 814, which appeared to be a mixture of petrolatum and rosin, thinned with some kerosene. This compound was given a rating of perfect in the exposure test. A mixture based on the formula given below was made up in the laboratory in an attempt to duplicate the sample above mentioned.

20 g rosin, "H" grade,
100 g petrolatum (U. S. P.),
10 cc kerosene.

The rosin was melted and mixed with the previously heated petrolatum and the resulting material thoroughly stirred. After the mixture had partially cooled the kerosene was added. The presence of the rosin greatly increased the adhesive property of the mixture over that of the original petrolatum.

Bright steel plates were coated with this material in the usual way and exposed to the weather on September 9, 1919. On May 5, 1920, the plates were brought into the laboratory and cleaned off by means of cotton waste wet with kerosene. The film of rust preventive was readily removed and the surface of the steel underneath was found to be smooth and practically free from rust. The plates were nearly as bright as when they were coated and exposed eight months before.

The results of the above exposure test indicate that a good slushing oil can be made from ordinary petrolatum and rosin. Where a rust preventive might be exposed to extreme hot weather, such as a temperature of 120° F met with in the sun's direct rays, it was thought best to add something to the petrolatum-rosin mixture to raise its melting point.

The U. S. P. petrolatum used was smeared over the bulb of a thermometer and heated in an air bath. At 37° C. a drop of grease fell from the thermometer and this point was taken as the melting point of the material. Mixtures of rosin with petrolatum showed melting points little higher than the petrolatum alone. For example, 30 parts of rosin and 100 parts of petrolatum mixed together hot showed a melting point of 40° C by the above method. A wax, like carnauba, with a high melting point, was found to raise the melting point of petrolatum to a much more pronounced degree. For example, 40 parts of carnauba wax and 100 parts of petrolatum were mixed hot, and the melting point of the cold mixture found to be 79° C. In a similar way 40 parts of candelilla wax and 100 parts of petrolatum was found to melt at 59.6° C.

Two per cent of carnauba wax was found to raise the melting point of petrolatum to 52.5° C, 15.5 degrees higher than petrolatum alone.

Two formulas for a slushing grease of semisolid consistency are given below:

FORMULA A.—3 parts candelilla wax, 6 parts rosin, grade "H," 50 parts petrolatum (U. S. P.).

FORMULA B.—2 parts carnauba wax, 5 parts rosin, grade "H," 50 parts petrolatum (U. S. P.).

The above materials should be heated together at about 125° C until all the ingredients are melted and the mixture then thoroughly stirred and allowed to cool.

Both of the above mixtures were found to pass all the requirements for a slushing oil given in the proposed specification on page 18. The above mixtures were also applied to bright steel plates by flowing an excess of the melted grease over the surface. These plates were exposed to the weather and were found to be free from rust after several weeks' time.

The mixtures described under formula A and formula B are not claimed to be the best rust preventives which can be made to fulfill the requirements of the proposed specification, but are cited merely as examples of easily made compounds which were found to prevent rust in a satisfactory manner.

There are many manufacturers who employ different raw materials and make rust preventives of equal or better quality.

WASHINGTON, June 16, 1920.

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SULPHUR IN PETROLEUM OILS

BY

C. E. WATERS, Chemist
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SULPHUR IN PETROLEUM OILS

By C. E. Waters

CONTENTS

	Page
I. Introduction	4
II. Origin of sulphur in petroleum	4
III. Forms in which sulphur occurs	4
1. Free sulphur	4
2. Hydrogen sulphide	5
3. Organic sulphur compounds	5
(*a*) Thiophene	5
(*b*) The thiophanes	5
(*c*) Alkyl sulphides or thio ethers	6
(*d*) Alkyl hydrosulphides or mercaptans	6
(*e*) Carbon bisulphide	6
(*f*) Sulphonic acids	6
(*g*) Alkyl sulphates	6
IV. Identification and significance of sulphur compounds	7
V. Detection of sulphur and its compounds	8
1. Copper test	8
2. Sodium test	10
VI. Determination of sulphur	10
1. Methods employing oxidizing agents	10
(*a*) Carius method	10
(*b*) Nitric acid digestion	10
(*c*) Rothe method	12
(*d*) Eschka method	13
(*e*) Sodium peroxide method	13
2. Methods involving combustion in oxygen or air	13
(*a*) Sauer method	13
(*b*) Dennstedt method	14
(*c*) Graefe method	14
(*d*) Lamp method	15
(*e*) Bomb calorimeter method	15
3. Miscellaneous methods	16
(*a*) Heating with sodium	16
(*b*) Heating with copper and copper oxide	16
(*c*) Lead peroxide	16
(*d*) Sodium hypobromite and hydroxide	17
(*e*) Digestion with permanganate	17
(*f*) Digestion with hydrochloric acid	17
(*g*) Hydrogenation	18
4. New method for sulphur	18
(*a*) Preliminary work	18
(*b*) Description of new method	19
(*c*) Criticism of new method	21
(*d*) Sources of error	22
(*e*) Correction for occlusion	23
VII. Summary	26

I. INTRODUCTION

Crude petroleum, from whatever source, contains sulphur. In the early days of the industry in this country, little or no attention was paid to this. The Pennsylvania oils contained but small amounts of this element, and the lighter distillates held lower percentages of sulphur than the crudes from which they were obtained. If this had not been so, the petroleum oils would no doubt have come into popular favor much more slowly than they did. Not only do oils which contain much sulphur have a disagreeable odor, but the products of their combustion are irritating and corrosive.

When the Canadian oil fields, and soon afterward those in Ohio and the adjacent States, were tapped, the removal of the comparatively large amounts of sulphur they contained became at once a refinery problem of the first importance. It is an interesting coincidence that Frasch, who years later developed the process by which the immense deposits of sulphur in Louisiana and Texas are worked, devised the first commercially successful method of removing sulphur from petroleum.

II. ORIGIN OF SULPHUR IN PETROLEUM

Several different sources of the sulphur and sulphur compounds which occur in petroleum have been suggested.[1] If the theory that the oil is derived from decayed animal remains be accepted, no other source of the sulphur need be sought, because this element is an essential constituent of all proteids.

It has been suggested that gypsum and other sulphates may be reduced by the oil. The sulphides thus formed could, according to the conditions, yield hydrogen sulphide or free sulphur. A portion of the latter might be deposited in the rocks. Again, oil percolating through these rocks might take up sulphur which was deposited in earlier ages.

III. FORMS IN WHICH SULPHUR OCCURS

1. FREE SULPHUR

Free sulphur has been found in numerous Roumanian[2] oils, as well as in some from California[3] and Texas.[4]

[1] Steuart, J. Soc. Chem. Ind., **19**, p. 989, 1900; and Naphta, **10**, p. 27, 1902; Peckham, Proc. Amer. Phil. Soc., **37**, p. 137, 1898; Engler, Fortschritte naturw. Forschung, **1**, p. 297; Mabery, Petroleum, **1**, p. 516; **2**, p. 319; Höfer, Erdöl u. s. Verw., Ed. 2, pp. 82 and 141; Höfer, Sitzber. k. Akad. Wiss. Wien, Math.-Nat. Kl., III, Abt. 1.

Nearly all of the data on the occurrence of sulphur in petroleum, as well as many literature references, were obtained from Engler-Höfer, Das Erdöl, **1**, pp. 463 to 479.

[2] Bourqoui, Chem. Rev. Fett-Harz-Ind., **8**, p. 210, 1901; Kissling, Chem.-Ztg., **26**, p. 492, 1902.

[3] Peckham, Proc. Amer. Phil. Soc., **36**, p. 108; 1897.

[4] Richardson and Wallace, Eng. Min. J., **73**, p. 352, 1902; and J. Soc. Chem. Ind., **21**, p. 316, 1902; Thiele. Chem.-Ztg., **26**, p. 896, 1902.

2. HYDROGEN SULPHIDE

Hydrogen sulphide is produced by the decay of organic matter, particularly under anaerobic conditions, and it is a frequent constituent of volcanic gases and occurs dissolved in subterranean waters. It is not surprising, therefore, that it is found in crude petroleum from many sources.[5]

Petroleum which contains free sulphur or organic sulphur compounds gives off hydrogen sulphide when heated, sometimes even at moderate temperatures. This appears to be somewhat at variance with what seems to be a well-established fact that the higher-boiling distillates contain a higher percentage of combined sulphur than the more volatile ones.

3. ORGANIC SULPHUR COMPOUNDS

It is very difficult to separate from crude petroleum any of its constituent hydrocarbons in pure form. Because of the ease with which many of them break down when heated, it is even more difficult to isolate and identify individual sulphur compounds, or even classes of compounds. However, members of more than one class of compounds have been identified. The evidence is not always as convincing as one could wish, for some of the compounds have been detected in distillates or found in the residues from crude oil. The ease with which hydrogen sulphide is formed by heating the oil shows that changes must take place in the organic sulphur compounds to some extent. No doubt those who have done most work in this field realize most fully the difficulties and limitations.

(*a*) THIOPHENE and some of its homologs apparently occur in some crudes, and their presence in certain light distillates has been proved by means of the indophenine reaction.[6]

(*b*) THE THIOPHANES are an interesting class of compounds which were obtained from the acid tar of a Canadian petroleum.[7] Although little is known of their structure, the empirical formula, $C_nH_{2n}S$, and their chemical behavior seem to show that they contain a polymethylene ring. They react readily with bromine,

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[5] Vender, Riv. scient. industr., **27**, p. 14, 1895; and Chem.-Ztg. Rep., **19**, p. 61, 1895; Nawratil, Dingl. pol. J., **246**, p. 423, 1882; Bolley, Ibid., **169**, p. 124, 1863; Mabery, Proc. Amer. Acad., **31**, pp. 17 and 43, 1894; Richardson, J. Frank. Inst., **162**, p. 115, 1906; Thiele, Chem.-Ztg., **25**, pp. 175 and 433, 1901; Richardson and Wallace, J. Soc. Chem. Ind., **20**, p. 690, 1901; Kast and Künkler, Dingl. pol. J., **278**, p. 38, 1890; Peckham, Proc. Amer. Phil. Soc., **36**, p. 108, 1897; Rakusin, Untersuchung des Erdöls, p. 102.

[6] Krämer, Verh. Vereins Beförd. Gewerbe-Fl., **1885**, p. 296; Meyer and Nahnsen, Ber. d. Chem Ges., **18**, p. 217, 1885; Edeleanu and Filiti, Bull. soc. chim., Ser. 3, **23**, p. 384, 1900; Charitschkoff, J. Russ. Phys.-Chem. Soc., **31**, p. 655, 1889, and Chem.-Ztg. Rep., **30**, p. 476, 1906; Girard, Petroleum, **2**, No. 3, 1906, and J. du Pétrole, **1906**, p. 69.

[7] Mabery and Quayle, Proc. Amer. Acad., **41**, p. 89, 1905; and Amer. Chem. J., **35**, p. 404, 1906; Mabery, J. Soc. Chem. Ind., **19**, p. 508, 1900.

but are not unsaturated compounds, because the amount of bromine evolved as hydrobromic acid is equal to that held in combination. By oxidation with alkaline permanganate solution, the thiophanes are converted into the corresponding sulphones, $C_nH_{2n}SO_2$.

The eight thiophanes isolated ranged in composition from $C_7H_{14}S$ to $C_{18}H_{36}S$.

(*c*) ALKYL SULPHIDES, OR THIO ETHERS, $(C_nH_{2n+1})_2S$, have been reported in certain Ohio crudes. Ten members of the series, from methyl sulphide, $(CH_3)_2S$, to hexyl sulphide, $(C_6H_{13})_2S$, were isolated.[8]

Kast and Lagai,[9] who may not have had the same crudes as Mabery and Smith, failed to find any compounds of this series in Ohio oils. On the other hand, Charitschkoff [10] detected alkyl sulphides in naphtha from Grosny.

The alkyl sulphides yield crystalline addition products with mercuric chloride.

(*d*) ALKYL HYDROSULPHIDES, OR MERCAPTANS, apparently occur in the distillates from Baku oil.[11]

(*e*) CARBON BISULPHIDE has been detected in petroleum ether (boiling point 50 to 80°C), but not in kerosene.[12] Compounds of the general formula, $C(C_nH_{2n+2})S$, have been obtained from certain Canadian petroleums.[13] They are apparently alkyl derivatives of carbon bisulphide.

(*f*) SULPHONIC ACIDS, RSO_3H, are said to cause the turbidity which is sometimes observed when distillates are allowed to stand for a long time.[14] They can be removed by vigorous agitation with a solution of caustic alkali.

(*g*) ALKYL SULPHATES.—Perhaps most of the compounds commonly called sulphonic acids are in reality alkyl sulphates, $(C_nH_{2n+1})_2SO_4$, which are formed when the oils are treated with sulphuric acid during refining. The saturated hydrocarbons do not react with the acid, but the unsaturated ones can form these esters by direct addition. For instance,[15]

$$2C_nH_{2n} + H_2SO_4 = (C_nH_{2n+1})_2SO_4.$$

[8] Mabery and Smith, Ber. d. Chem. Ges., 22, p. 3303, 1889; and Amer. Chem. J., 13, p. 232, 1890.

[9] Dingl. pol. J., 284, p. 69, 1892; 292, p. 116, 1894.

[10] J. Russ. Phys.-Chem. Soc., 31, p. 655, 1899; Chem.-Ztg. Rep., 21, p. 203; Chem. Centrbl., 1899, II, p. 920.

[11] Kwjatkowsky, in Höfer, Erdöl u. s. Verw., 2d ed., p. 82.

[12] Hager, Dingl. pol. J., 183, p. 165; 1867.

[13] The reference given by Höfer could not be verified, or the statement traced to its source.

[14] Veith, Dingl. pol. J., 277, p. 567: 1890.

[15] Vohl, Dingl. pol. J., 216, p. 47, 1875; Heusler and Dennstedt, Z. angew. Chem., 17, p. 264, 1904; Heusler, Ber. d. Chem. Ges., 28, p. 498, 1895.

The true sulphonic acids, which have their sulphur directly combined with carbon, can not be hydrolyzed by boiling with water or with dilute acids or alkalies. By careful fusion with caustic alkali the sulphonic group can be replaced by hydroxyl, as in one commercial process for the manufacture of phenol:

$$C_6H_5SO_3H + KOH = C_6H_5.OH + KHSO_3.$$

The alkyl esters, on the other hand, can be hydrolyzed with comparative ease by boiling with dilute acids or alkalies:

$$(C_nH_{2n+1})_2SO_4 + H_2O = 2C_nH_{2n+1}.OH + H_2SO_4.$$

When heated, these esters break down and give sulphur dioxide and carbon, among other products. These are formed even at the moderate temperatures reached in determining the Maumené number.[16]

IV. IDENTIFICATION AND SIGNIFICANCE OF SULPHUR COMPOUNDS

With the possible exception of the "sulphonic" acids, there seems to be no attempt made to determine the nature and amount of the sulphur compounds in routine laboratory practice. The difficulty, or even impossibility, rather than a lack of interest, is probably responsible for this. The oil chemist must feel that not only the total amount of sulphur, but also the nature of the compounds in which that element occurs, influence the behavior of oils. This is particularly true of lubricating oils which are subjected to service conditions favorable to deterioration. Heating in contact with air naturally comes first to mind because of the ease with which so many sulphur compounds are oxidized.

A conclusive decision as to the real influence of sulphur compounds upon the behavior of lubricating oils can be made only by comparing a sulphur-free oil with the same oil to which definite amounts of known sulphur compounds have been added. It does not appear probable that such tests can be made in the near future. However, it might be desirable to collect data on oils that have deteriorated badly in service in order to learn whether they contain higher percentages of sulphur than other oils which have stood up longer under the same conditions.

In this connection it may be pointed out that sulphur and sulphur dioxide cause an increase in the amount of asphaltic matter which is formed when oils are heated in the air.[17]

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[16] See footnote 15, and also Waters, B. S. Tech. Papers, No. 73, p. 12; 1916.

[17] Holde and Eickmann, Mitth. kgl. Materialprüfungsamt, 1907, p. 145; and Z. angew. Chem., 20, pp. 1263 and 1923, 1907; Waters, B. S. Tech. Papers, No. 4, p. 9, 1911; and J. Ind. and Eng. Chem., 8, p. 814, 1911; Southcombe, J. Soc. Chem. Ind., 30, pp. 261–264, 1911.

Work done recently at this Bureau shows that of four oils which had "carbonization values," or percentages of asphalt formed by oxidation, of 0.02 or less, none contained more than 0.08 per cent of sulphur. Of five oils which had carbonization values of 0.34 or more, all contained at least 0.29 per cent of sulphur. From these few figures it would appear that no oil which contains much sulphur will have a low carbonization value.

Transformer oils are regarded as undesirable if they contain more than a very small proportion of sulphur, because this element is believed to attack the wires and also to increase the tendency to form sludge.

In specifications for fuel oils the maximum permissible percentage of sulphur is generally stated, because of the corrosive effect of the products of its combustion. This is even more important for kerosene, because the oxides of sulphur are irritating and may bleach fabrics in the home, as well as corrode metal objects. The kerosene itself usually has a disagreeable odor when it contains more than a very low percentage of sulphur compounds.

Sometimes the presence of sulphur is desirable, as in flotation oils and some cutting oils. In the first case it is essential that the particles of ore cling to the oil drops in order to become separated from the gangue. The metallic sulphides more easily become oily than the other minerals. This oiling effect is helped by the presence of sulphur, perhaps by its action on particles or surface films of oxide which are thus changed to sulphide.

The principal reason for using a cutting oil is to lubricate the chips so that they can slide off the tool easily. It has been claimed that sulphur causes the oil to cling better to the metal by forming a film of sulphide. If this be true, the film is of extreme tenuity, because the metal shows little sign of tarnishing, as a rule.

V. DETECTION OF SULPHUR AND ITS COMPOUNDS

1. COPPER TEST

Free sulphur, hydrogen sulphide, and perhaps other sulphur compounds, in oils can be detected by the formation of a dark spot of copper sulphide when two or three drops of the oil are left on the polished metal for a sufficient length of time. The "copper dish" test for gasoline is based upon this reaction. It is generally believed that the degree of darkening depends upon the amount of sulphur present. This is not true. A topped Mexican crude, which contained 4.05 per cent of sulphur, and which gave off streams of hydrogen sulphide when heated in a test tube, did not perceptibly darken copper in 72 hours. Even

when a strip of the metal was partly immersed in the oil and kept at 295 to 300° C for 2 hours, the lower portion was only slightly tarnished. The end of the strip which was not under the oil was, however, much blackened and covered with a varnish-like coating, which was probably formed by partial oxidation of the oil.

Although the copper test can not be relied upon to show the presence of all sulphur compounds in oils, it is a delicate means of detecting free sulphur and hydrogen sulphide. A series of solutions was made by dissolving recrystallized sulphur in a white, medicinal oil which did not darken copper and which was found to contain too little sulphur to detect by analysis. The strongest solution contained 0.100 per cent of free sulphur. By suitable dilution, samples containing down to 0.0001 per cent, or 1 : 1 000 000, of sulphur were prepared. On each piece of freshly sandpapered, washed and dried piece of copper was placed two or three drops of oil. A blank test was run to check the original oil as well as the air of the laboratory. With 0.10 to 0.050 per cent of sulphur the copper was blackened in a few minutes, with 0.033 to 0.017 per cent the copper was perceptibly darker in 30 minutes, and with 0.01 per cent inside of an hour. When left in contact with copper for 20 hours, oil containing as little as 0.0002 per cent of sulphur, or 2 : 1 000 000, caused a just perceptible darkening of the metal. At the final dilution no action could be detected.

Similar tests were made with oil into which hydrogen sulphide had been passed for a few minutes. This solution, which instantly darkened copper, contained 0.037 per cent of sulphur, equivalent to 0.039 per cent of hydrogen sulphide. A portion of this was diluted to 100 volumes and then three or four times to 10 volumes each. The results of the tests with hydrogen sulphide are given in Table 1.

TABLE 1.—Copper Test for Hydrogen Sulphide

Dilution	Sulphur	Parts per million	Odor	Action on copper in 20 hours
Volumes	Per cent			
100	0.00037	3.7	Strong	Slight.
1 000	.000037	.37	Positive	Doubtful.
10 000	.0000037	.037	Very faint	None.
100 000	.00000037	.0037	None	Do.

It is evident that the copper test is much less delicate than the sense of smell.

2. SODIUM TEST

If the oil is not too volatile, but can be heated to 250 to 300° C without boiling out of the test tube, the sodium test can be applied. A test tube containing a few cubic centimeters of the oil is clamped in an inclined position over a flame which is regulated so that the oil is near its boiling point. A clean piece of sodium, 2 or 3 mm in diameter, is dropped in before the heating begins. After half an hour or longer, the oil is allowed to cool and is then poured off. The blackened lump of sodium is rinsed off with benzene, and dissolved in a few drops of water or 50 per cent alcohol. To the alkaline solution there are now added a few minute particles of sodium nitroprusside. If any sodium sulphide is present, the solution becomes pink to reddish purple, according to the amount. When only traces of sulphur are present, care must be taken not to mask the reaction by adding too much nitroprusside. Hydrogen sulphide does not produce a colored compound, so that the solution must not be acidified.

Attempts to utilize the sodium method for the determination of sulphur were unsuccessful, because duplicate results varied too widely.

VI. DETERMINATION OF SULPHUR

1. METHODS EMPLOYING OXIDIZING AGENTS.

(*a*) CARIUS METHOD.[18]—Although capable of giving highly accurate results, few chemists employ it when another can be used. It is not only troublesome and dangerous, but it has the disadvantage that only a few decigrams of material can be used in a determination. It is hardly applicable to oils low in sulphur. Even with oils that contain high percentages, there is danger of getting low results, owing to incomplete decomposition of the oil. Even when no more oily drops are to be seen, the oxidation may not be complete, as will be discussed more fully further on.

In the discussion of the Graefe method (see p. 14), some results obtained by the procedure of Carius are given.

(*b*) NITRIC ACID DIGESTION.—An obvious simplification of the Carius method is to digest the oil with fuming or concentrated nitric acid at atmospheric pressure, in a Kjeldahl flask or other suitable apparatus. There is no danger of explosions, and when the oil is low in sulphur, a large enough amount to yield a convenient weight of barium sulphate can be taken for analysis.

[18] Liebig's Ann., **116**, p. 1, 1860; **136**, p. 129, 1865; Ber. d. chem. Ges., **8**, p. 697, 1870.

But the oxidation of the oil proceeds very slowly, so that it may require 2 or 3 days to completely decompose as many grams. In order to hasten the reaction, potassium chlorate has been used.[19] According to the last authors cited, the results on California oils agree well with those obtained by the Carius method.

In this laboratory the use of chlorate was abandoned some years ago because it was found that permanganate hastened the oxidation of the oil. This is due to the formation of manganese peroxide, which spreads through the acid and comes into intimate contact with the oil.

The addition of a few drops of bromine, before the nitric acid is poured into the flask, seems to accelerate the reaction. At any rate it lessens the danger of losing hydrogen sulphide, if the oil contains any.

About a year ago it was found that, although duplicate determinations of the sulphur content of two Mexican oils agreed closely, when the nitric acid-permanganate method was used, yet the results were far too low. By the method described farther on, one of the oils was found to contain 1.83 per cent of sulphur.[20] The other, a topped Tampico crude, contained 4.05 per cent. Analyses by the method just described gave the following results for the two oils: First sample, 0.81, 0.80, 0.81, 0.87, 0.81, and 0.90 per cent; second sample, 2.17, 2.27, and 2.21 per cent.

It was noticed that even when all of the sample appeared to be decomposed, there was always some turbidity and usually oily drops separated out when the contents of the flask were washed into a beaker. This was due to the formation of nitrocompounds which were easily soluble in nitric acid but much less so in water. Although but slightly soluble in water, it was not possible to extract the nitroproduct with any reasonable amount of ether, and it was volatile with steam. The oily product dissolved in sodium-carbonate solution, with which it formed a dark brown salt; it was reprecipitated by acidifying the solution.

The nitrocompounds contained a considerable percentage of sulphur.[21] Failure to decompose the oil completely during the

[19] Kast and Lagai, Dingl. pol. J., 284, p. 71, 1892; Gilpin and Schneeberger, Amer. Chem. J., 50, pp. 65–66, 1913.

[20] This was a topped crude mixed with mineral seal. It was distributed for cooperative analysis by F. R. Baxter, chairman of the subcommittee on sulphur, committee D–2, American Society for Testing Materials. With his permission data taken from his report have been used in the preparation of this paper.

[21] According to Kast and Lagai (see footnote 19) the "resin" formed by the action of nitric acid and chlorate contains no sulphur.

digestion with acid and permanganate, together with its volatility and solubility, explains why low results for sulphur were obtained. The close agreement between the results actually found is in part accidental, in part due to working with nearly the same amounts of oil and reagents each time, and to stopping the digestion as soon as all oily drops disappeared.

In one determination with nitric acid and permanganate, the digestion was carried on in an all-glass apparatus so constructed that permanganate could be added from time to time. The escaping gases were passed through a solution of permanganate. This soon became decolorized by the oxides of nitrogen, but no sulphate was found in it. In this determination the oil was boiled for 25 hours with the acid, and permanganate was added from time to time. The percentage of sulphur found in the acid was 2.21, as against 2.17 and 2.27 in Kjeldahl flasks.

Goetzl,[22] in a similar experiment with fuming nitric acid, passed the escaping gases through caustic potash solution and found no sulphur.

He also heated fuel oil with fuming nitric acid, until it was all oxidized, then evaporated off the acid, extracted the solid mass with concentrated soda solution, acidified, and filtered off the "bituminous" matter which separated. The amount of sulphuric acid found in the filtrate was too low, but he seems to have missed the point that part of the sulphur is held in the nitrocompounds of which the residue is largely composed. As shown above, and confirmed by Goetzl's statement, this forms a salt with soda and is precipitated by hydrochloric acid.

(*c*) Rothe Method.[23]—The oil is digested in a flask with fuming nitric acid to which a little magnesium oxide has been added. After the acid has boiled for a time it is evaporated off, then more is added and the digestion is continued. This treatment is repeated a few times and finally any oil which remains is burned off with the aid of the magnesium nitrate. The residue in the flask is taken up with hydrochloric acid and water, the solution is filtered, and barium sulphate is precipitated.

The method has not been tested in this laboratory. It has been used elsewhere for the analysis of vulcanized rubber, although it is stated that duplicate results on compounds containing about 7.5 per cent of sulphur may be expected to differ by 0.2 or 0.3 per cent, and they may differ by 0.5 per cent.[24]

[22] Z. angew. Chem., 18, pp. 1528 to 1531; 1905.
[23] Described by Holde in Examination of Hydrocarbon Oils, English translation, pp. 40–41.
[24] Delft Rubber Inst. Communications, Part V, p. 144; 1917.

On the other hand Holde says that the method gave satisfactory results at the Materialprüfungsamt with oils containing no naphtha or illuminating oil.[25] None of these light fractions had been analyzed at the time he wrote.

(*d*) ESCHKA METHOD.—Originally devised for the determination of sulphur in coal,[26] the method has been applied also to the analysis of asphalt and oils. The sample is mixed with sodium carbonate and magnesium oxide and ignited in a crucible. The residue is treated with bromine water and nitric acid to oxidize all sulphur compounds to sulphuric acid, which is then precipitated as barium sulphate.

It has not seemed worth while to try the method, because of the certainty of losing sulphur as hydrogen sulphide or as volatile organic compounds. This seems to be confirmed by cooperative analyses of three samples of oil.[27] One was a heavy red oil known to contain sulphonic acids, the second a topped Mexican crude mixed with mineral seal, and the third a "300" oil which had been vulcanized by heating with sulphur. The last had a strong odor of hydrogen sulphide.

In one laboratory where Eschka's method was used, the results for the Mexican oil were 1.33 and 1.74 per cent. Five determinations at this Bureau gave from 1.80 to 1.85 per cent.

Two laboratories which used the Eschka procedure for the red oil found from 0.28 to 0.41 per cent. This Bureau found 0.51 to 0.59 in 8 determinations.

The figures on the vulcanized oil, as found in three laboratories, varied from 0.91 to 1.19 per cent; at this Bureau from 1.24 to 1.37 per cent in 8 analyses.

The method used by the Bureau is described further on.

(*e*) SODIUM PEROXIDE METHOD.—In recent years sodium peroxide has been used to a great extent for the determination of the halogens and sulphur. In 1895 Edinger employed it for the estimation of sulphur in volatile organic compounds.[28] The method has not been used for petroleum oils in this laboratory.

2. METHODS INVOLVING COMBUSTION IN OXYGEN OR AIR.

(*a*) SAUER METHOD.[29]—The sample of oil is slowly heated in a combustion tube while oxygen and carbon dioxide are supplied from opposite ends at such a rate that the oil vapors burn at a

[25] The Examination of Hydrocarbon Oils, English translation, p. 40.
[26] Oesterr. Ztschr. Berg- u. Hüttenwesen, **22**, p. 111; Z. anal. Chem., **13**, p. 344, 1874.
[27] See footnote 20, and Goetzl. Z. angew. Chem., **18**, p. 1258; 1905.
[28] Z. anal. Chem., **34**, p. 362; 1895.
[29] Z. anal. Chem., **12**, pp. 32–36; 1873.

constriction at the middle of the tube. The oxides of sulphur are absorbed in bromine water and barium sulphate is finally precipitated. Even with constant attention it is difficult to keep the flame where it should be and to prevent explosions in the tube, or else the escape of unburned oil. If the oil is low in sulphur, so large a sample must be taken that an analysis requires a long time.

DENNSTEDT METHOD.[30]—The chief feature of the Dennstedt procedure for the combustion of organic substances is the use of a catalyzer in the tube ahead of the boat containing the sample. Platinized quartz or a disk of the sheet metal is commonly used. When determinations of sulphur in oils were first made at this Bureau, the Dennstedt method, with platinized quartz, was used. Because only the sulphur was to be determined, the products of combustion were passed through a Meyer bulb tube containing sodium-hypobromite solution. After repeated trials the method was abandoned because, in spite of constant attention, it was almost impossible to prevent explosions or the escape of unburned gases. Another reason was the long time spent in making a combustion. About two working days was required to burn a 3 g sample. This included the time necessary to heat the catalyzer to redness, to cool the furnace at the end of the first day, and for all the other incidental operations.[31]

(*c*) GRAEFE METHOD.[32]—A small quantity of oil on absorbent cotton is burned in a large (7 l.) bottle of oxygen, in which is some sodium-hydroxide solution to absorb the oxides of sulphur. After the combustion the bottle is rinsed out, the solution is warmed with bromine and acidified, before adding barium chloride. Owing to the great heat developed, only about 0.3 g of oil can be burned, so that the method is not applicable to material low in sulphur.

Two fuel oils were analyzed by the Graefe and Carius methods in the laboratory of the Texas Co., and by a new procedure at this Bureau. Four determinations were made on each oil by each of the first two methods, and but two each by the new one.

The values by the new method were 0.54 and 0.53 per cent for one oil, and 0.76 and 0.77 per cent for the other. By the Carius method the figures varied from 0.62 to 0.65 and from 0.82 to 0.88,

[30] Ber. d. chem. Ges., 30, pp. 1590–1597; 1902.

[31] In determining nitrogen in petroleum by a combustion method Mabery (J. Amer. Chem. Soc., 41, p. 1697; 1919) found that "1 g of oil requires two or three hours in safe operation."

[32] Z. angew. Chem., 17, p. 616; 1904. See also Hempel. Ibid., 5, p. 393; 1892.

respectively; by the Graefe procedure, 0.56 to 0.66 and 0.84 to 0.89 per cent of sulphur.

In commenting on these analyses, the chemist of the Texas Co. said that he regarded the results of the three methods as in satisfactory agreement for oils which contained over 0.50 per cent of sulphur.

(*d*) LAMP METHOD.—The sulphur content of kerosene can be determined by burning it in a lamp and drawing the products of combustion through suitable absorption apparatus containing hypobromite solution.[33] The oil must burn with an ample supply of air and without a smoky flame. The air must be free from sulphur compounds. In some unpublished work by the author it was found that reducing gases are present in the products of combustion, and in greater amount the lower the flame. The lamp method for sulphur requires a rather low flame to prevent excessive heating of the absorption apparatus, so that the danger of losing organic compounds containing this element must be borne in mind.

Lubricating oils can be burned in a lamp if they are first mixed with light solvents.[34] According to Conradson, the sulphonic acids remain behind on the wick and can be determined separately. In this connection it should be mentioned that Heusler and Dennstedt claim that the charring of the wick is largely due to the presence of neutral alkyl esters of sulphuric acid. When they break down this acid is formed.[35]

(*e*) BOMB CALORIMETER METHOD.—As early as 1899 the bomb calorimeter was used for the determination of sulphur in oils.[36] Since then it has come to be the procedure most generally employed and the one in which most confidence is placed. The oil is burned in oxygen under high pressure, in a bomb calorimeter which contains a few cubic centimeters of water or of soda solution, or some sodium peroxide.

Even this apparently ideal procedure has certain disadvantages, apart from the cost of the apparatus. Not much more than 1 g of oil can be burned at a time and if the percentage of sulphur is low, very little barium sulphate will be obtained. If a lead gasket is used, it will be appreciably attacked by the oxides of sulphur, which will cause low results. Owing to the high tem-

[33] Heusler, Z. angew. Chem., 1895, p. 285; Engler, Chem.-Ztg., 20, p. 197, 1896.
[34] Albrecht, Diss. Karlsruhe, p. 37, 1907; Conradson, J. Ind. and Eng. Chem., 4, p. 842, 1912; and 8th. Int. Cong. Appl. Chem., 27, p. 19.
[35] Z. anorg. Chem., 17, pp. 264–5; 1904.
[36] Filiti, Bull. soc. chim., Ser. 3, 21, p. 338, 1899; and Z. anal. Chem., 39, p. 727, 1900.

perature of the combustion, some of the sulphur is burned only to the dioxide. If the bomb is opened too soon this can be tasted in the escaping gases. Under some conditions sulphur trioxide is absorbed slowly by water. A current of air carrying it can be bubbled through water without losing all of its cloudiness.

In the cooperative work referred to above (see p. 13) the three oils were analyzed by the bomb method in several laboratories. The extreme percentages of sulphur were:

	Per cent.
Mexican oil	1.52 to 1.90
Red oil	.39 to .53
Vulcanized oil	.89 to 1.22

One laboratory alone reported five determinations on the red oil which ranged from 0.42 to 0.53 per cent, while eight results for the vulcanized oil varied from 0.89 to 1.19 per cent.

The results obtained at this Bureau are given in the discussion of the Eschka method (see p. 13).

3. MISCELLANEOUS METHODS

In this section are described some attempts to find methods for total sulphur or for the differentiation of the sulphur compounds.

(*a*) HEATING WITH SODIUM.—Although heating with sodium to a comparatively high temperature seems to be a reliable way to test for the presence of sulphur, it could not be depended upon for quantitative data. The sodium was treated with water after the oil was removed with benzene, then bromine water and hydrochloric acid were added, the solution was filtered and finally barium sulphate was precipitated. The results varied greatly.

Metallic calcium under the same conditions appeared to remove no sulphur.

(*b*) HEATING WITH COPPER AND COPPER OXIDE.—Many experiments were made in which the oil was heated with copper powder or with the partially oxidized metal. The powder was prepared by sifting zinc dust into a solution of copper nitrate. The copper was thoroughly washed, finally with alcohol, and dried at a moderate temperature. For some of the tests this powder was heated until it became covered with oxide. The procedure was the same as for sodium, except that the residue, after washing with benzene, was dissolved in dilute hydrochloric acid containing bromine. Considerable sulphur was removed from the oil by this treatment, but the results rarely checked one another.

(*c*) LEAD PEROXIDE.—This reagent was found to remove very little sulphur when heated with oils. In this case the residue was

extracted with sodium-carbonate solution, as recommended by Dennstedt, or with sodium hypobromite.

(*d*) SODIUM HYPOBROMITE AND HYDROXIDE.—These reagents were used in attempts to remove at least a definite part of the sulphur from the Mexican oil which contained 1.83 per cent of sulphur. A solution of hypobromite, made by adding 1.5 cc of bromine to 50 cc of a cold, 5 per cent caustic soda solution, was shaken with 2.2 to 2.3 g of oil at frequent intervals for three and one-half hours. The mixture was then warmed on the steam bath for one and one-half hours and allowed to stand over night. The aqueous layer was separated and acidified. The addition of barium chloride produced merely traces of precipitate, although the smallest sample taken contained enough sulphur to yield over 290 mg of barium sulphate.

A 5 per cent solution of caustic soda, when heated on the steam bath with the oil and frequently shaken for several hours, extracted only insignificant amounts of sulphur. In this case bromine was added before the hydrochloric acid and barium chloride.

(*e*) DIGESTION WITH PERMANGANATE.—When 0.5 g samples of the Mexican oil which contained 4.05 per cent of sulphur were digested on the steam bath with a strong solution of potassium permanganate for several hours some sulphur was oxidized. In two determinations the amounts of barium sulphate were equivalent to 0.30 and 0.37 per cent, respectively, of sulphur. There is no way, at present, of deciding whether this represented a general attack upon all of the sulphur compounds in the oil, or whether only a particular group of such derivatives was oxidized.

(*f*) DIGESTION WITH HYDROCHLORIC ACID.—This work was done chiefly in order to detect esters of sulphuric acid—the "sulphonic acids," as they are often called. Ten-gram samples of the heavy red oil (see p. 13), which contained about 0.55 per cent of sulphur, were digested on the steam bath with hydrochloric acid of three dilutions, from 1:2 to 2:1. Other portions were boiled with 1:5 acid and still others heated with 1:4 acid in sealed tubes. These were hung inside of a steam bath for several hours. In nine determinations the amounts of sulphuric acid found varied from 0.063 to 0.105 per cent, which corresponds to 0.020 to 0.034 per cent of sulphur.

In the analysis of turkey red oil and other "sulphonated" oils, the usual procedure is to boil with hydrochloric acid. In our tests digestion on the steam bath gave results a few thousandths

of a per cent higher than those obtained by boiling or by heating in sealed tubes. It is not necessary to boil if the flasks are frequently shaken.

Besides the red oil, the two Mexican oils were also tested. The negligible amounts of sulphuric acid obtained from 10 g samples were equivalent to only 0.001 and 0.004 per cent of sulphur, respectively.

(*g*) HYDROGENATION.—An attempt was made to remove sulphur by heating oil with hydrogen in the presence of a catalyzer. In the first test an engine oil which contained 0.85 per cent of sulphur was used. The apparatus was so arranged that a bulb holding the oil and the catalyzer was heated in an air bath made of an iron crucible with a perforated cover of asbestos board. A slow current of hydrogen bubbled through the oil, passed through a glass tube which was long enough to cool it, and escaped through a Volhard flask containing sodium-hypobromite solution.

The first catalyzer used was made by igniting nickel oxide in hydrogen. The reduced metal was pyrophoric, so that it changed back to oxide before it was mixed with the oil.

At first 3.225 g of oil, with 0.01 g of catalyzer, was heated for two and one-half hours at 320 to 330° C. The hypobromite solution yielded only 0.0023 g of barium sulphate out of a possible 0.1996 g. The same lot of oil was then heated to a much higher temperature, with the thermometer removed, for three hours, after which 0.0043 g more sulphate was obtained. The total of 0.0066 g corresponded to 3.3 per cent of the sulphur in the oil.

Because the topped Mexican crude which contained 1.83 per cent of sulphur gave off streams of hydrogen sulphide when heated, it was tested in the way just described. In five hours' heating, 1.9908 g yielded only 0.0017 instead of a possible 0.2653 g of barium sulphate. When a 7 mm circular disk of platinum foil was used as a catalyzer, 1.7984 g of oil gave 0.0046 g of sulphate.

In the experiments at the higher temperatures the thermometer was removed from the bath when 360° was approached and the flame was increased until the oil vapors condensed 5 or 6 cm above the cover. Only traces of oily distillate were carried over into the hypobromite.[37]

4. NEW METHOD FOR SULPHUR

(*a*) PRELIMINARY WORK.—In a sense all of the methods tested intermittently as opportunity offered since 1908, may be looked

[37] After this work was finished it was learned that Goetzl (Z. angew. Chem., 18, p. 1529; 1905) attempted to determine the sulphur content of fuel oil by distilling off hydrogen sulphide. The amount of this gas which was set free was not proportional to the quantity of oil distilled off.

upon as leading up to the one finally hit upon. Early in 1910 a series of comparative tests was made of variations of the Henriques method for the determination of total sulphur in rubber. According to the original procedure the rubber is digested with nitric acid, the excess of which is evaporated off. The residue is then oxidized by heating with a mixture of soda and saltpeter. The melt is dissolved in hot water, the solution filtered, acidified and treated with barium chloride. It was found that under certain conditions some sulphur, apparently that in the free state, escaped oxidation by the nitric acid and was lost when the latter was evaporated off. There was no such loss of sulphur when the rubber was treated at the start with nitric acid saturated with bromine.[38]

This success in retaining all of the sulphur in rubber goods led to the use of bromine for the preliminary attack of oils by the nitric acid-permanganate method. At that time, and indeed up to 1919, it was not thought worth while to try the procedure of Henriques for oils, because of the belief that much of the sulphur would be lost by volatilization in organic compounds. This is true if the oil is not first attacked by nitric acid, but it is now known that the nitrocompounds which are formed yield salts when treated with sodium carbonate, and the sulphur is retained.

An oil which six determinations by the nitric acid-permanganate method showed to contain from 0.80 to 0.90 per cent of sulphur, was incorporated with a mixture of soda and saltpeter and carefully ignited. The result was 1.37 per cent. When fused in the same way after treatment with nitric acid and bromine, five determinations gave values from 1.80 to 1.85 per cent. These were the first obtained by the method to be described. A proportionately close agreement is what has come to be expected of beginners in rubber analysis, because there are no complications in the technic.

(*b*) Description of New Method.—A preliminary paper describing the method has already been published.[39] The directions here given have been changed but little.

Weigh from 0.5 to 2.0 g of oil in a 100 cc porcelain crucible, add 5 cc of concentrated nitric acid which has been saturated with bromine, and at once cover with a watch glass. When rather volatile oils, or those which smell of hydrogen sulphide are ana-

[38] Waters and Tuttle, B. S. Bulletin, 8, pp. 445–453, 1911; B. S. Scientific Papers No. 174; J. Ind. and Eng. Chem., 3, Oct., 1911. See also Tuttle and Isaacs, B. S. Tech. Papers, No. 45, 1915.

[39] Waters, J. Ind. and Eng. Chem., 12, pp. 482–485 and 612, 1920.

lyzed, the crucible should be covered during the weighing, and each sample should be treated with the acid at once, and not be left until the series of weighings is finished. Place on the steam bath, but not in direct contact with the steam, and digest for 30 minutes or more, with an occasional gentle swirling of the contents of the crucible. Unless the mixture threatens to foam up to the cover, the crucible may now be heated by the direct steam for two or three hours. If there is much foaming, which happens but seldom, the digestion at moderate heat must be continued longer.

Remove the crucible from the bath, and when its contents have cooled somewhat, add cautiously, in small portions, 10 or 12 g of anhydrous sodium carbonate. This is conveniently measured in a 15 cc crucible, rather than weighed. The first portions of soda often remain on top of the oily layer, so that the crucible must be tilted or swirled slightly to start the reaction. When about half of the soda has been added, rinse the watch glass with 1 or 2 cc of warm water and leave it off. Pour in the rest of the soda. With a glass rod, about 3 mm thick and 12 cm long, thoroughly mix the contents of the crucible and spread the pasty mass around on the sides and half to two-thirds of the way to the top. The rod is left in the crucible, which is then placed on the steam bath, or in an air bath at 100° C.

When its contents are dry enough, the crucible is placed in an inclined position on a wire triangle and the ignition is started over a low flame. When more than 1 g of oil has been taken, there is a tendency for it to burn too briskly, but this is easily controlled by judicious use of the stirring rod which scrapes the burning portion away from the rest. When part of the mass has burned white, more is worked into it, and so on until all of the organic matter is destroyed. It is necessary to hold the crucible with tongs during this operation. Toward the end of the ignition the flame may be increased somewhat, but it is rarely necessary to heat the crucible to redness. With care a good crucible should last for many determinations. Of two which were used for 17 determinations each, and were still in almost perfect condition, one crucible lost just 100 mg, and the other 1.1 mg less.

If a suitable muffle is available, the ignitions can be made at a temperature not above 550°. The glass rod should be removed and the crucible covered with a watch glass. The principal difficulty is that the oil may start to burn at some point and the whole mass begin to glow in a few seconds. In this case the cover

may be blown aside and loss by spattering occur. Under proper conditions, several ignitions can be made at one time.

According to the amount of oil taken, the mixture of sodium salts resulting from the combustion varies from granular to pasty when hot. After cooling, place the crucible upright in a 400 cc beaker and fill it with distilled water. After a short time lay it on its side, cover the beaker and digest on the steam bath. When the salts have dissolved, remove and rinse the crucible. Filter the solution to remove specks of carbon, bits of the glass rod, etc. Acidify with concentrated hydrochloric acid, using Congo red paper as indicator, and add 2 cc of acid in excess. The neutralization proceeds more safely if nearly the required amount of acid is put in the covered beaker into which the solution is filtered. It is safe to use 10 cc of acid for this, but the exact amount can not be stated, because the total amount of carbonate and nitrite to be neutralized depends on how much oil was taken. The acidified filtrate and wash water should have a volume of about 175 cc. Heat on the steam bath, add 10 cc of barium-chloride solution (containing 100 g of crystallized salt in 1 l), and digest on the bath until the supernatant liquid is clear. If possible delay the filtration until the next day.

A blank should be run with every new lot of reagents. An ignition is made just as if oil were present, because the flame may contain enough sulphur to be corrected for. Even when the correction amounts to several milligrams almost no precipitate may be seen before the solution has stood for several hours.[40]

It is needless to describe how the barium sulphate is filtered off, washed, and ignited.

(*c*) Criticism of New Method.—It is an advantage that several determinations can be started at one time. The digestions and filtrations for these can be carried on simultaneously. An ignition requires 15 to 25 minutes. (Rubber is so completely broken down that less time is spent in this part of the procedure.) Oil is but slightly decomposed by the treatment with acid, and most of it is driven off in clouds of vapor during the first part of the ignition, and it appears as if much of the sulphur were escaping also. The concordance of duplicate results and their sub-

[40] It is not necessary to have the solution boiling when the barium chloride is added, or to digest the precipitate with the boiling solution. Hillebrand (Analysis of Silicate and Carbonate Rocks, Bull. 700, U. S. Geol. Surv., p. 232) says "at boiling heat or on the steam bath." It is much more convenient to use the steam bath, and there is no danger of loss by bumping; also, as pointed out by Hillebrand, there can be no contamination by sulphur from the flame.

stantial agreement with analyses by the bomb method are evidence that there can be little or no loss.

In discussing the bomb method a few comparative figures were given. The following were obtained in the analysis of five fuel oils in which the sulphur had been determined by the Bureau of Mines laboratory at Pittsburgh.[41] Each oil was analyzed twice by the new method. The figures in parentheses are the values obtained by the bomb method at the Bureau of Mines. The percentages of sulphur were: 3.67, 3.70 (3.17); 4.01, 4.03 (3.98); 1.13, 1.12 (1.01); 0.36, 0.36 (0.29); 0.18, 0.17 (0.13).

Whatever the faults of the new method, it gives results at least as high as those by the bomb. It has been criticized as too bothersome, yet it does not require the same meticulous attention to details as when the bomb is used. For 10 years it has been used at this Bureau for the analysis of rubber, and even in the hands of beginners has given good results. For oils it may well be employed in laboratories which do not have sufficient work of this kind to justify the expense of a bomb calorimeter.

(*d*) SOURCES OF ERROR.—The most obvious way in which incorrect results can be obtained—failure to convert all of the sulphur into sodium sulphate—appears not to be a fault of this method. It has, however, one serious fault in common with all of the other procedures in which salts or oxides of metals are used to effect or facilitate the oxidation of the sulphur compounds in the oil. This is the occlusion of the salts of these metals by the barium-sulphate precipitate. Even when barium chloride is added to water which contains only sulphuric acid, the precipitate is not pure, but is contaminated by more or less chloride, and the results are too high. If soluble sulphates, particularly those of sodium, potassium, or ammonium, are present, there is a considerable amount of occlusion and during the ignition of the precipitate a complication of reactions, with the net result that the values found are too low. On the other hand, the presence of nitrates causes errors in the opposite direction. Finally, the amount of dissolved barium sulphate which is lost in the filtrate and washings depends on their volume and on the quantity of free acid, other than sulphuric.[42]

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[41] They were obtained through the interested cooperation of E. W. Dean and N. A. C. Smith, the latter in the Washington laboratory of that Bureau.

[42] For an interesting discussion of the determination of sulphur as barium sulphate, see the following papers; Allen and Johnston, J. Am. Chem. Soc., **32**, pp. 588–617, 1910; Johnston and Adams, Ibid., **33**, pp. 829–845. 1911.

(*e*) CORRECTION FOR OCCLUSION.—By adapting to the present purposes a suggestion made by Johnston and Adams in the paper just cited, data were obtained which show the necessary corrections of weights of barium sulphate up to 0.200 g. In order to have definite amounts of sulphur, a solution of pure sodium sulphate which was dried at 200° C before weighing out, was prepared. By taking various aliquot portions of this solution, weights of barium sulphate from 3 to 200 mg were obtained. Without going into all the details, the procedure was as follows: To check the purity of the sodium salt an aliquot portion was diluted to 175 cc, acidified with 2 cc of concentrated hydrochloric acid, heated on the steam bath and precipitated with 10 cc of the usual barium-chloride sulution. The results were in satisfactory agreement with those calculated.

In an actual determination of the sulphur in oil, the aqueous solution of the melt contains sodium carbonate, nitrate, nitrite, and sulphate. When this is acidified, the carbonate and nitrite are decomposed, so that finally only chloride, nitrate, and sulphate are present. The amount of chloride depends indirectly upon the weight of oil taken. This is true also of the residual nitrate. In order to have approximately the same conditions as when amounts of oil varying from 0.5 to 2.0 g were burned, portions of sodium carbonate equal to those used in an analysis were dissolved and treated with 2, 3, or 4 cc of concentrated nitric acid. Then to the solution was added hydrochloric acid until there was 2 cc in excess beyond the neutral point. The desired aliquot portion of sulphate solution was added, the volume made up to 175 cc, and barium sulphate precipitated as usual. Next day the precipitates were filtered off and finally weighed. Blanks were run under the same conditions, only with the sodium sulphate left out. The small amounts found in the blanks were used as corrections for the larger weights of barium sulphate.

No matter how much sodium sulphate was taken, the weights of barium sulphate found, after being corrected for the impurities in the reagents, were greater than those calculated from the weights of sodium sulphate. The differences between the weights found and those calculated were the corresponding corrections for occlusion and solubility. On plotting these corrections against the weights of barium sulphate found, it was seen that a straight line running from the origin to a point with the coordinates 10 and 200 mg represented the facts with sufficient accuracy. In

other words, the net error amounted to 5 per cent of the weight of the precipitate. The variations from this straight line were so slight as to be of no significance in the analysis of oils. The most surprising fact was that changing the volume of nitric acid caused so little difference in the amount of occlusion.

With a 1 g sample, 200 mg is equivalent to 2.75 per cent of sulphur and the corrected weight, 190 mg, to 2.61 per cent. Under other conditions of volume, acidity, etc., the corrections, and the errors if these are neglected, might be very different.

An easy way to find the weights of sulphur corresponding to the amounts of barium sulphate found is by means of a graph. Figure 1 is such a graph drawn from the data obtained in the determinations of the occluded salts. Each centimeter on the axis of abscissas represents 10 mg of barium sulphate. On the axis of ordinates each centimeter represents 1 mg of sulphur. After correction, 200 mg of barium sulphate becomes 190 mg, which corresponds to 26.1 mg of sulphur. From the point represented by the coordinates 200 and 26.1 draw a straight line to the origin. For any weight of sulphate up to 200 mg the weight of sulphur can be read off directly, and its percentage calculated. If the same weight of oil, say 1 g, is taken for analysis each time, the percentages, instead of the weights of sulphur can be laid off on the axis of ordinates; thus instead of 26.1 mg there would be 2.61 per cent. It is not always desirable to use so little or so much as 1 g of oil, and it may be advisable to make one calculation rather than to delay long enough to make an exact weighing if the oil is losing hydrogen sulphide. For these reasons it seems preferable to lay off the ordinates as first stated.

An example from an actual analysis is given: 0.5053 g of oil yielded 0.0722 g of barium sulphate. The sodium carbonate was so poor that the correction for it was 0.0085 g. This subtracted from the first weight gave 0.0637 g. On the graph 63.7 mg of barium sulphate is seen to be equivalent to 8.3 mg of sulphur. This divided by 0.5053 gives 1.64 per cent. The percentage calculated without the graph is 1.66, an amply satisfactory agreement.

After the manuscript of the present paper was completed, and a few days before a preliminary paper was published in the Journal of Industrial and Engineering Chemistry (May, 1920, pp. 482 to 485), it was learned that an almost identical method was published 15 years ago. This earlier paper seems to have been entirely overlooked or ignored by oil chemists. Goetzl

(Z. angew. Chem., **18**, pp. 1528 to 1531; 1905) used the following procedure for the determination of sulphur in "liquid fuel": In a roomy platinum crucible treat 2 or 3 g of oil with 4 cc of fuming nitric acid. The crucible, covered with a watch glass, is allowed

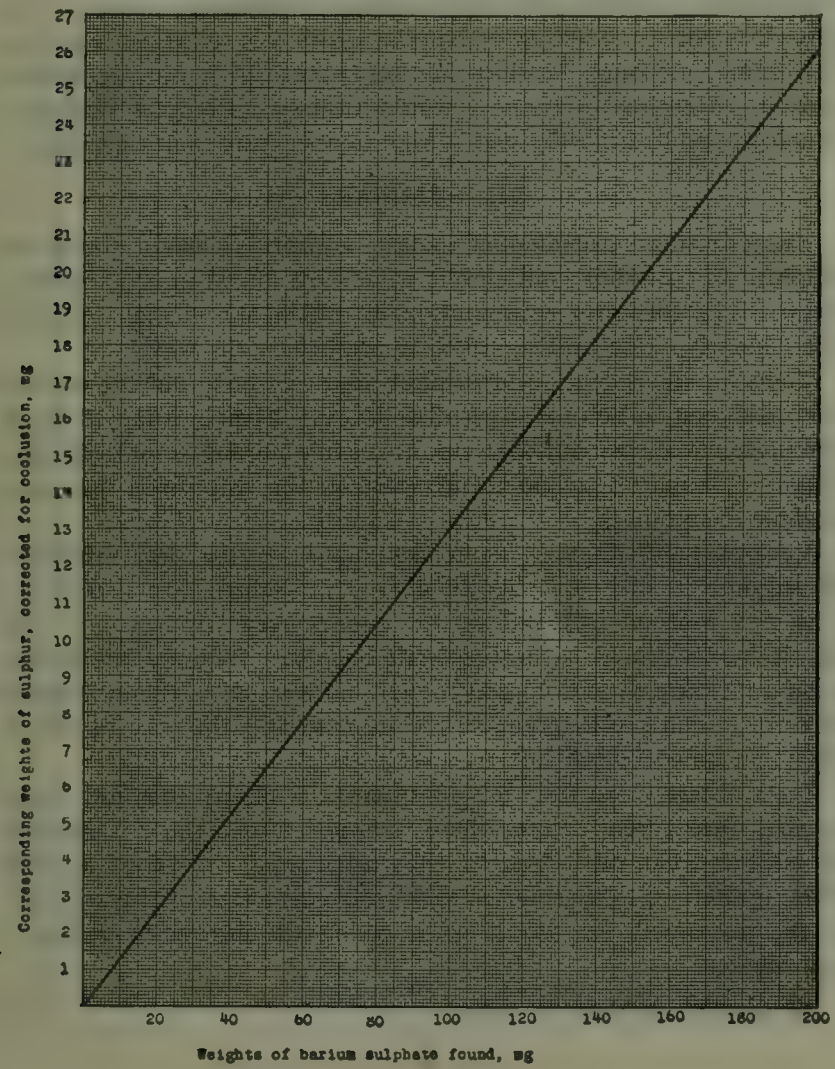

FIG. 1.

to stand over night and then warmed gently on a water bath. The acid is evaporated off and the oily nitrated mass is mixed with sodium carbonate and potassium nitrate. More of the fusion mixture is spread over the top and the whole is ignited. The subsequent operations need not be described.

Kerosene and naphtha contain so little sulphur that 10 g samples were taken for analysis. The method was used for coal, lignite, peat, organic substances, etc.

As in the majority of papers on the determination of sulphur, no mention of errors due to occlusion is made by Goetzl. He also does not say whether or not the ignition is to be made over a full flame, although he does advise the use of a mushroom-top burner. Heating to redness would rapidly destroy a porcelain crucible, and fortunately such a high temperature in not necessary.

VII. SUMMARY

Short accounts are given of theories concerning the origin of the sulphur and sulphur compounds which are found in crude petroleum. The forms of combination in which the element occurs, their identification and significance are briefly discussed.

Tests for the detection of sulphur are described, and the copper test is shown to be one of great delicacy. Although it will show the presence of very minute amounts of free sulphur or of hydrogen sulphide, it may be of no value when all the sulphur is in stable organic compounds.

Various methods that have been used for the determination of sulphur in oils, and finally a new procedure, are described. The new method is based on the preliminary treatment of the oil with nitric acid saturated with bromine, followed by fusion with a mixture of sodium nitrate and carbonate. Certain sources of error which are inherent in the method and the way in which they can be corrected for, are taken up.

Data obtained by the analysis of certain oils by this and other methods are given. From these it appears that there is no loss of sulphur when oil is treated with nitric acid and bromine, and then ignited with sodium nitrate and carbonate. The method is recommended for laboratories which do not have a bomb calorimeter.

WASHINGTON, May 28, 1920.

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STEEL RAILS FROM SINK-HEAD AND ORDINARY RAIL INGOTS

BY

GEORGE K. BURGESS, Physicist

Bureau of Standards

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By George K. Burgess

CONTENTS

	Page
I. General plan of investigation	5
II. Detailed scheme of procedure	6
1. Ingots	6
2. Blooms	16
3. Rails	17
4. Tests	19
III. Description of operations and discussion of results	19
1. Ingot practice	19
2. Temperature observations	20
3. General comparison of the two types of ingot	21
(*a*) Size of ingots	21
(*b*) Loss in heating	22
(*c*) Comparison by heats	22
(*d*) Inspection of ingots	28
(*e*) Comparison of chemical composition	28
(1) Sink-head ingots	28
(2) Comparison ingots	28
4. The sink-head ingots, their manufacture and classification	29
(*a*) Sink-head effect	29
(*b*) Piping in sink-head ingots	30
(*c*) Influence of surface condition of ingots on rail classification	30
5. The comparison ingots, their manufacture and classification	30
6. Comparison of chemical segregation of ingots of both types	32
(*a*) Segregation in sink-head ingots	32
(*b*) Segregation in comparison ingots	34
(1) Group M1–M5	34
(2) Group M6–M10	36
(3) Group M11–M15	36
(*c*) Segregation of elements other than carbon	41
7. Mechanical tests	41
(*a*) Method of expressing the results of mechanical tests	41
(*b*) Ultimate strength and yield point	43
(*c*) Elongation and reduction of area	47
(*d*) Hardness	48
(*e*) Drop test	48
8. Metallographic tests	49
9. Examination of split ingots and blooms	50
(*a*) Sink-head ingot H37	50
(*b*) Sink-head ingot H32	50
(*c*) Sink-head H5 and comparison M10 top blooms	55
10. Special nickel chromium sink-head ingot	55

Page

IV. Service of rails from this investigation 56
V. Summary and conclusions 59

LIST OF TABLES

1. Dimensions of ingots of both types 7
2. Results of chemical analyses and head particulars of sink-head ingots 8
3. Results of chemical analyses of Maryland steel 8
4. Results of inspection of sink-head ingots and blooms 9
5. Time consumed in rolling and results of inspection of comparison ingots and blooms 10
6. Pouring temperatures of comparison ingots 10
7. Temperatures of working of sink-head ingots 11
8. Temperatures of working of comparison ingots 11
9. Typical rolling temperatures 12
10. Disposition of material from sink-head ingots 12
11. Disposition of material from comparison ingots 15
12. Classification of rails from sink-head and comparison ingots 18
13. Mechanical and chemical characteristics of rails from sink-heads ingots 23
14. Mechanical and chemical characteristics of rails from comparison ingots 24
15. Percentage carbon segregation in rails from sink-head ingots 33
16. Percentage carbon segregation in rails from comparison ingots 33
17. Necessary total discard (percentage) in rails from sink-head ingots 34
18. Necessary total discard (percentage) in rails from comparison ingots 35
19. Percentage segregation of carbon in the "A" rails of sink-head ingots 37
20. Percentage segregation of carbon in the "A" and discard rails of comparison ingots 39
21. Total top discard required in sink-head ingots 42
22. Total top discard required in comparison ingots 42
23. Mechanical and chemical properties of the special sink-head (nickel and chromium) ingot 56

LIST OF FIGURES

1. Diagram showing method of cutting of blooms and rails from sink-head ingots. 17
2. Diagram showing method of cutting of blooms and rails from comparison ingots 17
3. 100-pound P. S. rail section showing location of various tests 17
4. Sink-head ingot 29
5. Photograph of sulphur print of top bloom from Maryland ingot No. 10 48
6. Photograph of sulphur print of top bloom from Hadfield ingot No. 5 48
7. Photograph of sulphur print of half section of Hadfield ingot No. 32 48
8. Rail M1–A from Maryland ingot 48
9. Rail M5–F from Maryland ingot 48
10. Rail M4–F from Maryland ingot 48
11. Rail H16–A from Hadfield ingot 48
12. Rail H16–E from Hadfield ingot 48
13. Comparison of ingots of both types; elastic limit—thousand pounds per square inch 43
14. Comparison of ingots of both types; ultimate strength 44
15. Comparison of ingots of both types; elongation 45
16. Comparison of ingots of both types; reduction of area 46
17. Comparison of ingots of both types; Brinell hardness 47
18. Diagram of Hadfield split ingot No. 32, showing percentage of carbon 51
19. Diagram of Hadfield split ingot No. 32, showing percentage of phosphorus .. 52

Page
20. Diagram of Hadfield split ingot No. 32, showing percentage of silicon..... 53
21. Diagram of Hadfield split ingot No. 32, showing percentage of sulphur..... 54
22. Diagram of top bloom from Hadfield ingot H5, showing chemical analysis of various positions..... 57
23. Diagram of top bloom from Maryland ingot M10, showing chemical analysis of various positions..... 58

I. GENERAL PLAN OF INVESTIGATION

The results of the experimental rolling into rails of several types of ingots, including several designs of sink-head ingots and others, with the cooperation of the Maryland Steel Co., reported by Hadfield and Burgess,[1] looked sufficiently promising to the rail committee of the Pennsylvania Railroad to warrant the purchase of 100 tons of ingots cast by Sir Robert Hadfield in England, in order to make a more effective study of the suitability of this type of sink-head ingot for the manufacture of rails. It was also considered desirable to carry out similar series of tests of ingots of ordinary rail steel rolled at the same time and under as nearly identical conditions as possible in the same mill.[2] President F. W. Wood, of the Maryland Steel Co., Sparrows Point, Md., offered the facilities of that plant for this investigation, as in the case of previous experiments reported by Hadfield and Burgess. The rolling of ingots for the present investigation was done in 1915 at the Sparrows Point plant. The reduction of the results has been greatly delayed, but it is thought the subject matter is still of sufficient interest to warrant publication.

The planning of the details of the investigation, after correspondence with Sir Robert Hadfield, was made by A. W. Gibbs, chief mechanical engineer of Pennsylvania Railroad Co.; F. W. Wood, president of Maryland Steel Co.; and Dr. G. K. Burgess, chief, division of metallurgy, Bureau of Standards; and the tests were carried out by members of the technical staffs of their respective organizations, to whom acknowledgment is here made for their indispensable aid.

It was expected by Sir Robert Hadfield that the sink-head ingots made under this system would yield rails of remarkably high quality as regards "soundness, freedom from all piping and all segregation." From an inspection of the data concerning the manufacture of the ingots made by this process he also felt con-

[1] Hadfield and Burgess, Sound Steel Ingots and Rails, Trans. Am. Inst. Min. Engrs., **51**, pp. 862–880, 1915; J. Iron and Steel Inst., **91**, pp. 40–124, 1915.

[2] See also the valuable paper comparing American-made sink-head and ordinary ingots for rails by Edward F. Kenney: The Commercial Production of Sound and Homogeneous Steel, Am. Iron and Steel Inst., p. 144, 1915.

fident in stating: "This shows in a remarkable manner the uniformity of the ingots; also that every one of them is perfectly sound, free from blowholes, with no piping, and segregation below 8 per cent discard."

The results of the present investigation of 37 ingots of the Hadfield type show that he was not far astray in the confident prediction of uniformity, physical soundness, and freedom from chemical segregation. In fact, it is difficult to imagine 37 ingots, from as many heats of steel, more uniform in quality and properties and as free from those undesirable qualities and uncertainties that beset the ordinary ingot from which rails are made.

The ingots with which they were compared, the product of three heats, 5 from each heat, or 15 ingots in all, are not less instructive, in that each group, or heat, represents a distinct practice in open-hearth manufacture. In this investigation, therefore, from the point of view of steel making, we are really comparing the products rolled from 2 types of ingot representing 4 methods of manufacture.

The rolling, inspection, tests, and analyses were carried out with the greatest care, and by a series of actual weighings all the material from the several ingots was accounted for.

II. DETAILED SCHEME OF PROCEDURE

The detailed scheme of observations of rolling and tests of sink-head and comparison ingots follows:

1. INGOTS

Method of Numbering Ingots.—The sink-head ingots were numbered H1, H2, etc., ... H37, and the ingots made in the ordinary manner were numbered M1, M2, etc., ... M15.

Weight of Ingots.—The sink-head ingots were weighed cold and the others were weighed hot. The approximate weight of the sink-head ingots was 5300 pounds each and of the comparison ingots 7300 pounds.

Size and Shape of Ingots.—The English-made ingots were cast with sink head on large end, while the American-made ingots were cast small end up; dimensions of both types of ingot are shown in Table 1, and details concerning the sink heads of Hadfield ingots are to be found in Table 2.

Composition of Ingots.—Each sink-head ingot was from a separate heat, the compositions of which, furnished by the manufacturer, are given in Table 2. The American-cast ingots were from three heats, and their chemical compositions are shown in Table 3.

TEMPERATURE OF INGOTS.—The temperature of pouring of the comparison ingots is shown in Table 6; the temperature of the sink-head ingots was taken when they were introduced into the soaking pits, the temperatures of which were also noted then and at intervals until the ingots were withdrawn from the soaking pits; the temperature of English (Table 7) and comparison (Table 8) ingots were also taken at the blooming mill. In Table 9 are shown typical ingot-rolling temperatures of both types of ingot.

INSPECTION OF INGOTS.—The sink-head ingots were inspected cold for surface defects, cracks, etc., and at the blooming mill during rolling for the existence and location of cracks, scrap, etc. (Table 4). The other ingots were similarly inspected at the blooming mill (Table 5).

DISPOSITION OF INGOTS.—Of the 37 sink-head ingots cast at Sheffield, 35 were rolled into blooms at the Sparrows Point mill, and the remaining 2 ingots were sectioned longitudinally for metallographic examination. The examination of H32 was made by the Bureau of Standards and that of H37 by Hadfields (Ltd.). In addition to splitting 2 ingots, it was also decided to examine similarly top blooms from one of each of the two types of ingot, H5 and M10. The blooms and ingots examined in this manner were selected at random. All blooms were rolled into rails directly without reheating, and all material was accounted for by actual weighing, as shown in Tables 10 and 11.

TABLE 1.—Dimensions of Ingots of Both Types

Part measured	Sink-head ingot	Comparison ingot
	Inches	Inches
Top	19½ by 19½	18¼ by 18¼
Bottom	17¼ by 17¼	19¼ by 19¼
Height	[a] 50½	63

[a] The height given is exclusive of the height of sink head, which was approximately 13¾ inches.

TABLE 2.—Results of Chemical Analyses and Head Particulars of Sink-Head Ingots [a]

Ingot number	Heat number	Chemical composition (percentages)					Sink-head particulars			
		C	Si	S	P	Mn	Height to which filled in head	Depth of settling	Capacity of cavity	Percentage of settling to total bulk of ingot
							Inches	Inches	Cm^3	
H1	4774	0.65	.....	0.040	.....	0.94	14.00	8.25	[illegible]	3.19
H2	4738	0.63–.64	.....	.042	.....	.91	13.50	9.00	[illegible]	3.31
H3	4847	.63–.64	.....	.047	.....	.93	13.75	9.25	10450	3.37
H4	4915	.65–.66	.....	.049	.....	.92	14.00	9.75	9675	3.10
H5	4873	.69	.....	.050	.....	.95	14.00	9.38	10250	3.29
H6	4939	.63–.64	0.16	.045	0.028	.92	13.00	7.50	8800	2.84
H7	4627	.64	.....	.052	.....	.97	14.00	8.25	9600	3.08
H8	[illegible]	.65	.14	.049	.029	.92	13.75	8.50	10250	3.29
H9	4709	.58	.....	.049	.....	.93	13.00	7.75	[illegible]	2.88
H10	[illegible]	.63–.64	.16	.042	.031	.86	13.25	9.25	[illegible]	3.52
H11	5018	.64	.16	.039	.031	.92	14.00	8.75	10040	3.21
H12	4641	.67–.69	.....	.045	.....	.92–.94	14.50	8.50	[illegible]	2.89
H13	5059	.63–.64	.....	.049	.....	.92	14.00	8.88	10750	3.44
H14	4835	.69	.....	.046	.....	.91	13.50	7.75	9520	3.05
H15	4978	.67–.68	.....	.051	.....	.96	14.50	9.88	[illegible]	2.95
H16	4992	.66	.....	.045	.....	.94	13.50	9.75	[illegible]	2.88
H17	4825	.66	.....	.045	.....	.92	13.50	6.88	[illegible]	2.89
H18	4829	.66	.....	.041	.....	.98	14.00	8.50	11100	3.55
H19	5073	.63–.64	.....	.045	.....	.90	14.00	9.50	[illegible]	3.37
H20	[illegible]	.66	.....	.038	.....	.96	13.50	6.50	8600	2.77
H21	[illegible]	.66	.....	.036	.....	.95	13.50	8.13	9150	2.90
H22	4723	.63	.....	.044	.....	.95	13.50	8.00	9900	3.17
H23	5046	.62–.63	.....	.042	.....	.93	13.50	7.00	[illegible]	3.37
H24	5030	.60	.....	.056	.....	.94	13.50	10.13	11950	3.82
H25	[illegible]	.58	.....	.042	.....	.93	14.50	8.50	10100	3.24
H26	4813	.64	.14	.048	.032	.93	14.00	8.63	[illegible]	3.45
H27	4615	.64	.20	.048	.....	1.01	14.25	7.00	[illegible]	2.89
H28	4588	.64	.19	.047	.....	.97	13.00	7.25	8930	2.86
H29	4908	.64	.....	.041	.....	.84	14.00	8.75	9750	3.11
H30	4861	.61–.62	.21	.045	.031	.92	14.00	9.63	10900	3.49
H31	[illegible]	.65	.15	.049	.....	.96	13.50	8.50	9825	3.16
H32	[illegible]	.65	.....	.052	.....	.94	14.00	8.50	10175	3.27
H33	4761	.65	.....	.048	.....	.90	13.50	8.50	[illegible]	3.47
H34	[illegible]	.63–.64	.....	.039	.....	.86	11.00	8.88	10040	3.34
H35	[illegible]	.63	.....	.050	.....	.89	14.00	9.00	10500	3.36
H36	4653	.61	.....	.053	.....	.85	13.50	7.75	9900	3.17

[a] Furnished by Sir Robert Hadfield.

TABLE 3.—Results of Chemical Analyses of Maryland Steel

Heat number	Ingot numbers	Elements determined (percentages)						
		C	Si	S	P	Mn	Cr	Ni
2 x 4497	M1 –M5, inc.	.608–0.630	0.148	0.067	0.010	0.710–0.720	0.270	0.760
1 x 3632	M6 –M10, inc.	.642– .654	.185	.092	.014	.650– .660	.290	.780
2 x 4510	M11–M15, inc.	.642– .650	.191	.097	.019	.760– .810	.270	.760

TABLE 4.—Results of Inspection of Sink-Head Ingots and Blooms

Ingot number	Results of inspection of cold ingots for cracks, scrap, etc.	Time of charging ingots into pits	Time of drawing ingots from pits	Interval in pits	Time consumed in rolling in blooming mill	Results of inspection during rolling in blooming mill
					Min. sec.	
24	1 side—bad splash in pouring, 1 rough corner	4:35 p. m. to 4:40 p. m., Aug. 15, 1915.	11:56 a. m. to 12:25 p. m., Aug. 16, 1915.	19 hrs., 32 mins.	2 10	Very small piece of scrap on one corner.
28	1 side—bad splash in pouring bottom half, 1 rough corner				2 22	Very small piece of scrap on sides and corners.
2	1 side rough, 2 rough corners				2 25	1 crack 18 inches from top on side of ingot.
15	1 side very rough near top, 2 rough corners				2 15	2 small cracks on corners 2 feet from top of ingot.
1	Sides good, 2 rough corners				2 10	Spot of scrap 2 feet from top.
21	1 rough side, 1 rough corner				2 20	Spot of scrap 2 feet from top. Small crack on corner 2 feet from top.
9	3 rough sides, 1 rough corner	4:45 p. m. to 4:52 p. m., Aug. 15, 1915.	12:29 p. m. to 12:46 p. m., Aug. 16, 1915.	19 hrs., 49 mins.	2 25	Cracked all around ingot at bottom of sink-head.
7	Sides O. K., 2 rough corners				2 15	Small spot of scrap 8 inches from top on corners.
35	3 sides—rough spots 1 foot from bottom, 1 rough corner				2 35	Small spots of scrap all over ingot.
13	3 rough sides, 2 rough corners				2 20	Small spots of scrap 12 inches from bottom.
22	1 side—bad splash in pouring, 1 rough corner				2 10	Small spots of scrap on corners 2 feet from bottom.
14	2 sides splashed in pouring, 3 rough corners				2 20	Clean bloom.
19	1 side—bad splash in pouring, 2 rough corners	4:30 p. m. to 4:38 p. m., Aug. 16, 1915.	1:49 p. m. to 2:05 p. m., Aug. 17, 1915.	21 hrs., 22 mins.	1 30	Small spot of scrap on corner 1 foot from top.
26	1 side rough 1 inch from top, 2 rough corners				2 15	Small spots of scrap 2 feet from top.
18	1 side—bad splash in pouring, corners good				2 15	Cracked at bottom of sink-head.
6	2 sides—bad splash in pouring, 2 rough corners				2 20	1 small spot of scrap 4 feet from top and one 1 foot from bottom on sides.
23	2 sides—bad splash in pouring, 1 rough corner				2 25	
31	1 side—bad splash in pouring near bottom, 2 rough corners				2 30	1 spot of scrap 1 foot from top on side.
						Clean bloom.
						Clean bloom.
29	1 side—crack in mold 10 inches from top, 2 rough corners.	4:50 p. m. to 5:05 p. m., Aug. 16, 1915.	2:06 p. m. to 2:22 p. m., Aug. 17, 1915.	21 hrs., 15 mins.	2 40	Spots of scrap on corners and sides 18 feet from top.
	2 sides—rough spots in center				.. ..	
36	2 sides—rough spots 1 foot from bottom, corners good				2 20	Small spot of scrap 2 feet from top.
4	4 sides—splash in pouring, 2 rough corners				2 15	Spots of scrap at center across the corners.
11	2 sides—splash in pouring, 2 rough corners				2 20	Small spots of scrap all over the top third of ingot.
12	1 side—splash in pouring, corners good				2 25	Small spots of scrap 2 feet from top on corners and sides.
10	Sides good, 3 rough corners				2 30	Small spots of scrap 3 feet from bottom on corners.
16	2 sides—rough 2 feet from bottom, 1 rough corner	4:30 p. m. to 4:45 p. m., Aug. 17, 1915.	9:51 a. m. to 10:10 a. m., Aug. 18, 1915.	17 hrs., 22 mins.	2 20	Small spot of scrap 1 foot from top on corner.
20	2 sides rough, 3 rough corners				2 15	Clean bloom. Cracked at bottom of sink-head.
33	3 sides bad splash in pouring 1 foot from top, 1 rough corner				2 20	Small spot of scrap at center on sides.
17	1 side—splash in pouring, 2 rough corners				2 20	Large spots of scrap on all sides.
30	3 sides—splash in pouring at center, 2 rough corners				2 15	Small spots of scrap 2 feet from top on sides.
27	1 side—splash in pouring 2 feet from bottom, 1 rough corner				2 30	Small spots of scrap on all sides.
						Cracked at bottom of sink-head.
8	1 side—splash in pouring 6 inches from bottom, 1 rough corner.	4:45 p. m. to 5:00 p. m., Aug. 17, 1915.	10:14 a. m. to 10:27 a. m., Aug. 18, 1915.	17 hrs., 28 mins.	2 25	Large spot of scrap at center of ingot on one side.
3	2 rough sides, 2 rough corners				2 40	Small spot of scrap 2 feet from top.
25	3 sides—splash in pouring, 1 rough corner				2 20	Large spot of scrap 2 feet from top on corner.
34	1 side rough at center, 2 rough corners				2 50	Large spots of scrap on all sides.
5	1 side—splash in pouring, corners good				2 25	Small spots of scrap on sides.
32	1 side—splash in pouring, 2 rough corners				.. ..	Not rolled.

TABLE 5.—Time Consumed in Rolling and Results of Inspection of Comparison Ingots and Blooms

Ingot number	Date rolled	Heat number	Time in molds	Time in soaking pits	Time consumed in rolling in blooming mill	Results of inspection during rolling in blooming mill
			Min.	H. M.	M. S.	
1	Aug. 16, 1915	2x4497	20	2 10	2 25	1 corner and 1 side cracked 1 foot from bottom.
2	do	2x4497	20	2 14	1 50	Small spot of scrap 2 feet from top on corner.
3	do	2x4497	20	2 18	2 35	Clear bloom.
4	do	2x4497	20	2 21	2 ..	Do.
5	do	2x4497	20	2 23	2 ..	Do.
6	Aug. 18, 1915	1x3632	23	1 40	1 50	Two small side cracks 4 feet from bottom.
7	do	1x3632	23	1 40	1 45	Clear bloom.
8	do	1x3632	23	1 41	2 10	Do.
9	do	1x3632	23	1 43	1 55	Do.
10	do	1x3632	23	1 45	2 10	1 small side crack 5 feet from bottom.
11	do	2x4510	30	2 15	2 ..	Clear bloom.
12	do	2x4510	30	2 15	2 ..	Small side crack 3 feet from top.
13	do	2x4510	30	2 17	2 ..	Clear bloom.
14	do	2x4510	30	2 19	2 10	Small side crack 3 feet from top.
15	do	2x4510	30	2 20	2 5	Small side crack 2 feet from bottom.

TABLE 6.—Pouring Temperatures of Comparison Ingots [a]

Observation number	Temperature for heat 1x3632	Temperature for heat 2x4510	Observation number	Temperature for heat 1x3632	Temperature for heat 2x4510
	°C	°C		°C	°C
1	.....	1557	16	1543	1522
2	.....	1545	17	1533	1512
3	[b] 1596	[c] 1545	18	1533	1507
4	[b] 1587	[c] 1538	19	1539	1497
5	[b] 1564	[c] 1540	20	1523	.....
6	[b]	[c] 1528	21	1539	.....
7	[b] 1578	[c] 1531	22	1537	.....
8	1559	1532	23	1537	.....
9	1557	1537	24	1529	.....
10	1557	.....			
11	1549	1527			
12	1557	1531			
13	1547	1521			
14	1551	1533			
15	1539	1527			

[a] Pouring temperatures group M_1–M_5, inclusive, not taken.
[b] Ingots from group M6–M10, inclusive. (Average pouring temperature, 1581°C.)
[c] Ingots from M 11–M15, inclusive. (Average pouring temperature, 1536°C.)

TABLE 7.—Temperatures of Working of Sink-Head Ingots

Ingot number	Temperature of soaking pits		Ingot temperature		Rail temperature at finishing pass (taken on base of rail)	
	At time of charging ingots	After 14½ hours	When drawn	Blooming mill	First rail bar	Second rail bar
	°C	°C	°C	°C	°C	°C
1	700	1310	1300	1130	1016	1007
2	700	1310	1300	1140	1030	1020
15	700	1310	1300	1137	1026	1017
21	700	1310	1300	1123	1025	1005
24	700	1310	1300	1172	1025	1010
28	700	1310	1300	1157	1019	1008
7	750	1300	1290	1125	1028	998
9	750	1300	1290	1149	1024	1016
13	750	1300	1290	1116	1034	1008
14	750	1300	1290	1143	1043	1019
22	750	1300	1290	1144	1032	1018
35	750	1300	1290	1140	1034	1011
6	650	1200	1295	1149	1048	1025
18	650	1200	1295	1168	1032	1023
19	650	1200	1295	1180	1069	1044
23	650	1200	1295	1154	1047	1026
26	650	1200	1295	1173	1047	1034
31	650	1200	1295	1146	1036	1010
4	650	1240	1290	1156	1040	1016
10	650	1240	1280	1147	1046	1022
11	650	1240	1280	1136	1029	1012
12	650	1240	1280	1142	1047	1029
29	650	1240	1290	1148	1034	1005
36	650	1240	1280	1136	1038	1016
16	600	1305	1515	1182	1018	955
17	600	1305	1515	1164	1036	1016
20	600	1305	1515	1179	1045	1023
27	600	1305	1515	1153	1040	1024
30	600	1305	1515	1157	1033	1020
33	600	1305	1515	1160	1029	1017
3	600	1300	1316	1156	1031	1023
5	600	1300	1316	1149	.....	.....
8	600	1300	1316	1151	1029	1033
25	600	1300	1316	1153	1035	1000
34	600	1300	1316	1140	1014	1004
Average	658	1276	1299	1150	1034	1014
Maximum	750	1310	1315	1182	1069	1044
Minimum	600	1200	1280	1116	1014	955

TABLE 8.—Temperatures of Working of Comparison Ingots

Ingot number	Ingot temperature in blooming mill	Rail temperature at finishing pass (taken on base of rail)	
		First rail bar	Second rail bar
	°C	°C	°C
1	1149	1026	1005
2	1173	1016	993
3	1164	1040	1025
4	1179	1053	1039
5	1177	1064	1027
6	.....	1031	1025
7	1167	1029	1018
8	1176	1030	1041
9	1159	1038	1009
10	1180	1038	.....
11	1162	1039	1008
12	1181	1045	1025
13	1161	1032	1018
14	1149	1025	1015
15	1149	1024	1018
Average	1164	1035	1001
Maximum	1181	1064	1039
Minimum	1149	1016	993

TABLE 9.—Typical Rolling Temperatures

Temperature of sink-head ingot number							Temperature of comparison ingot number					
7	13	14	19	28	30	34	2	5	7	10	13	15
°C	°C	°C	°C	°C	°C	°C	°C	°C	°C	°C	°C	°C
1136	1157	1152	1212	1181	1195	1165	1184	1173	1179	1206	1179	1168
1147	1114	1147	1212	1171	1179	1165	1171	1184	1179	1198	1171	1157
1144	1103	1136	1176	1152	1165	1157	1168	1176	1176	1177	1168	1136
1114	1109	1147	1176	1147	1147	1141	1184	1176	1160	1179	1157	1149
1123	1109	1141	1160	1147	1147	1131	1171	1179	1163	1173	1157	1147
1125	1128	1141	1165	1147	1139	1123	1165	1184	1157	1168	1147	1136
1112	1093	1139	1165	1147	1144	1120	1163	1179	1157	1163	1147	1144
1103	.....	.....	.....	.....	1141	1125	.....	1147	.....	.....	.....	.....
Average							Average					
1125	1116	1143	1180	1157	1157	1141	1173	1177	1167	1180	1161	1149

	°C		°C
Grand average	1145	Grand average	1168
Maximum	1212	Maximum	1206
Minimum	1093	Minimum	1136

TABLE 10.—Disposition of Material from Sink-Head Ingots

[Weight in pounds. Discard made only to physically sound steel. H_5 not included in this table as the top bloom for this ingot was sent to the Bureau of Standards]

Ingot number	Ingot weight	Bloom crops				Weight of the four test pieces each 6 inches long	Rail crops at hot saw		
		Top		Bottom			Middle		Bottom
		Weight	Per cent of ingot, average	Weight	Per cent of ingot, average		No. 1	No. 2	
ROLLED AUG. 16, 1915									
24	5300	185	3.5	297	5.6	86	77	60	259
28	5300	233	4.4	112	2.1	79	74	32	[illegible]
2	5300	155	2.9	113	2.1	79	66	30	21
15	[illegible]	167	3.2	129	2.4	79	[illegible]	31	48
1	5300	190	3.4	123	2.3	77	[illegible]	[illegible]	30
21	5300	442	8.4	116	2.2	[illegible]	43	77	75
9	5300	294	5.5	131	2.5	79	46	75	89
7	5250	234	4.5	122	2.3	79	[illegible]	[illegible]	74
35	5300	280	5.3	161	3.0	77	[illegible]	[illegible]	50
13	5300	191	3.6	141	2.7	81	[illegible]	[illegible]	[illegible]
22	5250	437	8.3	123	2.3	[illegible]	47	45	54
14	[illegible]	231	4.4	138	2.6	78	53	[illegible]	67
ROLLED AUG. 17, 1915									
19	5300	160	3.0	120	2.3	77	[illegible]	50	69
26	5300	160	3.0	130	2.5	75	47	53	70
18	5300	237	4.5	116	2.2	76	50	77	[illegible]
6	5300	324	6.1	127	2.4	77	44	56	[illegible]
23	5350	134	2.5	131	2.4	78	49	79	100
31	5300	234	4.4	109	2.1	74	54	28	[illegible]
29	5350	566	10.6	123	2.3	77	44	63	78
36	5300	342	6.5	142	2.7	76	50	47	71
4	5350	270	5.0	123	2.3	71	50	[illegible]	56
11	5300	325	6.1	136	2.7	73	73	39	61
12	5350	204	3.8	124	2.3	74	54	60	97
10	5250	320	6.1	130	2.5	78	46	57	[illegible]
ROLLED AUG. 18, 1915									
16	5350	199	3.7	154	2.9	74	37	56	[illegible]
20	[illegible]	276	5.2	122	2.3	73	43	66	72
33	5300	228	4.3	148	2.8	75	49	78	[illegible]
17	5300	145	2.7	117	2.2	74	53	73	[illegible]
30	5300	247	4.7	112	2.1	74	45	79	117
27	5300	245	4.7	97	1.9	[illegible]	57	[illegible]	71
8	[illegible]	142	2.7	119	2.3	78	53	75	[illegible]
3	5300	205	3.8	115	2.2	71	81	55	[illegible]
25	[illegible]	160	3.0	151	2.8	77	[illegible]	57	72
34	5300	195	3.7	166	3.2	[illegible]	[illegible]	43	57
Total	179 700	8357	4.3	4519	2.5	2623	[illegible]	[illegible]	2540

TABLE 10—Disposition of Material from Sink-Head Ingots—Continued

ROLLED AUG. 16, 1915—Continued

Ingot number	Ingot weight	Weight of rails				Total weight rolled, including crops and test pieces	Loss	
		A	B	C	D		Weight	Per cent of ingot, average
24	5300	762	1111	1117	1122	5082	218	4.1
28	5300	1232	1112	1120	1121	5145	155	2.9
2	5300	1315	1119	1120	1123	5141	159	3.0
15	5300	1277	1115	1120	1123	5172	128	2.4
1	5300	1284	1116	1122	1123	5162	138	2.6
21	5300	941	1121	1121	1124	5145	155	2.9
9	5300	1096	1121	1117	1119	5167	133	2.5
7	5250	1203	1116	1119	1122	5177	73	1.4
35	5300	1172	1113	1115	1118	5180	120	2.3
13	5300	1289	1116	1118	1120	5211	89	1.7
22	5250	1032	1116	1117	1122	5174	76	1.4
14	5300	1228	1113	1115	1117	5190	110	2.1

ROLLED AUG. 17, 1915—Continued

19	5300	1175	1108	1112	1115	5035	265	5.0
26	5300	1226	1108	1110	1112	5091	209	3.9
18	5300	1130	1107	1111	1115	5118	183	3.4
6	5300	1035	1107	1111	1111	5061	239	4.5
23	5350	1183	1102	1105	1109	5070	280	5.2
31	5300	1191	1100	1110	1117	5066	234	4.4
29	5350	867	1107	1111	1111	5147	203	3.8
36	5300	1082	1106	1106	1113	5135	165	3.1
4	5350	1228	1106	1107	1111	5168	182	3.4
11	5300	1161	1107	1116	1114	5205	95	1.8
12	5350	1194	1101	1107	1114	5129	221	4.1
10	5250	1077	1110	1114	1115	5108	142	2.7

ROLLED AUG. 18, 1915—Continued

16	5350	1191	1111	1111	1115	5127	223	4.2
20	5300	1175	1107	1111	1113	5158	142	2.7
33	5300	1153	1107	1112	1112	5155	145	2.7
17	5300	1269	1107	1113	1115	5159	141	2.7
30	5300	1151	1111	1112	1113	5161	139	2.6
27	5300	1229	1119	1120	1121	5200	100	1.9
8	5300	1274	1117	1109	1112	5165	135	2.5
3	5300	1204	1103	1106	1109	5114	186	3.5
25	5300	1275	1104	1109	1112	5181	119	2.2
34	5300	1157	1105	1115	1113	5092	208	3.9
Total	179 700	39 464	37 749	37 859	37 946	174 791	5519	3.0

TABLE 10.—Disposition of Material from Sink-Head Ingots—Continued

ROLLED AUG. 16, 1915—Continued

Ingot number	Ingot weight	Discard in rail on account of—			Material discarded to physically sound steel		Material available for rails			Ingot rolled with no draft on
		Sink head	Pipe	Total	Weight	Per cent of original ingot, average	Weight	Per cent of original ingot, average	Net per cent of ingot, average	Pass number
24	5300	161	.....	161	346	6.5	4043	76.3	87.8	1
28	5300	163	.....	163	396	7.5	4501	84.9	89.2	1
2	5300	230	.....	230	385	7.3	4526	85.4	88.9	1
15	5300	310	.....	310	477	9.0	4404	83.1	88.1	2
1	5300	292	.....	292	482	9.1	4430	83.5	87.8	2
21	5300	162	.....	162	604	11.1	4230	79.8	84.8	2
9	5300	.....	.....	.....	294	5.5	4532	85.5	91.8	1
7	5250	235	.....	235	469	8.9	4404	83.9	88.2	1
35	5300	160	.....	160	440	8.3	4435	83.7	88.6	1
13	5300	80	.....	80	271	5.1	4644	87.6	92.7	2
22	5250	.....	.....	.....	437	8.3	4468	85.1	88.8	2
14	5300	284	.....	284	515	9.7	4367	82.4	97.5	2

ROLLED AUG. 17, 1915—Continued

19	5300	326	.....	326	486	9.2	4261	80.4	85.0	1
26	5300	233	.....	233	393	7.4	4323	83.0	87.9	1
18	5300	.....	324	324	561	10.5	4215	79.5	85.0	1
6	5300	158	.....	158	482	9.1	4283	80.8	85.5	2
23	5350	291	.....	291	425	7.9	4286	80.1	86.7	1
31	5300	237	.....	237	471	8.9	4355	82.2	86.0	2
29	5350	64	.....	64	630	11.8	4209	78.7	84.3	1
36	5300	73	.....	73	415	7.8	4410	83.2	88.4	1
4	5350	87	162	249	519	9.7	4374	81.8	87.0	1
11	5300	254	.....	254	579	10.9	4317	81.5	86.4	2
12	5350	367	.....	367	571	10.7	4223	78.9	85.1	2
10	5250	148	.....	148	468	8.9	4346	82.8	86.8	2

ROLLED AUG. 18, 1915—Continued

16	5350	305	.....	305	504	9.4	4297	80.4	86.4	1
20	5300	313	.....	313	589	11.1	4266	80.5	85.4	1
33	5300	281	.....	281	509	9.6	4278	80.7	86.7	1
17	5300	342	.....	342	487	9.1	4336	81.8	87.4	2
30	5300	270	.....	270	517	9.8	4291	80.9	86.8	2
27	5300	233	.....	233	478	9.0	4434	83.7	88.5	2
8	5300	346	.....	346	488	9.1	4344	82.0	87.5	1
3	5300	378	.....	378	583	11.0	4215	79.5	84.6	1
25	5300	340	.....	340	500	9.4	4337	81.8	87.5	1
34	5300	389	.....	389	584	11.0	4184	78.9	84.1	2
Total	179 700	7512	486	7998	16 355	9.1	147 643	81.9	87.9	.....

TABLE 11.—Disposition of Material from Comparison Ingots

[Weights in pounds. Discard made only to physically sound steel]

HEAT NO. 2x4497

Ingot number	Ingot weight	Bloom crops				Weight of the five test pieces, each 6 inches long
		Top		Bottom		
		Weight	Per cent of ingot, average	Weight	Per cent of ingot, average	
1	7360	183	2.4	276	3.7	90
2	7030	243	3.4	209	2.9	98
3	7040	185	2.6	143	2.0	100
4	7130	189	2.6	226	3.1	95
5	7190	223	3.1	175	2.4	101
	35 750					

HEAT NO. 1x3632

6	7340	189	2.5	291	3.9	93
7	7200	205	2.8	215	2.9	90
8	7260	188	2.5	215	2.9	90
9	7240	225	3.1	252	3.4	91
	29 040					

HEAT NO. 2x4510

11	7510	654	8.8	236	3.1	96
12	7340	288	3.9	254	3.4	89
13	7190	490	6.8	175	2.4	88
14	7320	379	5.1	210	2.8	95
15	7480	236	3.1	235	3.1	96
	36 840					
Total	101 630	3857	3.8	3112	3.0	1314

HEAT NO. 1x3632

10		3315		230		97

HEAT NO. 2x4497—Continued

Ingot number	Ingot weight	Rail crops at hot saw			Weight of rails					
		Middle		Bottom	X	A	B	C	D	E
		No. 1	No. 2							
1	7360	48	58	71	939	1117	1117	1117	1123	1126
2	7030	84	96	94	517	1114	1115	1117	1120	1122
3	7040	95	92	74	675	1110	1109	1115	1117	1119
4	7130	147	81	37	660	1108	1108	1115	1116	1120
5	7190	89	100	59	774	1108	1109	1113	1115	1119
	35 750									

HEAT NO. 1x3632—Continued

6	7340	44	83	75	878	1118	1118	1121	1121	1123
7	7200	34	56	25	880	1116	1114	1117	1122	1122
8	7260	60	59	73	888	1111	1112	1122	1120	1120
9	7240	25	59	94	783	1118	1118	1122	1123	1125
	29 040									

HEAT NO. 2x4510—Continued

11	7510	36	87	145	571	1102	1106	1111	1110	1116
12	7340	48	76	115	826	1105	1103	1106	1108	1111
13	7190	42	29	123	825	1101	1101	1104	1104	1106
14	7320	37	80	127	775	1099	1099	1104	1101	1103
15	7480	69	70	123	1011	1103	1106	1107	1102	1108
	36 840									
Total	101 630	856	1006	1238	10 813	15 530	15 535	15 591	15 609	15 640

HEAT NO. 1x3632—Continued

10			68	140				1115	1116	1117

TABLE 11.—Disposition of Material from Comparison Ingots—Continued

HEAT NO. 2x4497—Continued

Ingot number	Ingot weight	Total weight rolled, including crops and test pieces	Discard in rail on account of pipe	Material discarded to physically sound steel		Material available for rails			Date rolled
				Weight	Per cent of original ingot, average	Weight	Per cent of original ingot, average	Per cent of net ingot, average	
1	7360	7256	505	688	9.3	6115	83.1	88.5	Aug. 16, 1915
2	7030	6929	.....	243	3.4	6203	88.2	94.8	Do.
3	7040	6934	.....	185	2.6	6345	90.1	95.8	Do.
4	7130	7002	336	525	7.4	5936	83.9	90.2	Do.
5	7190	7085	.....	223	3.1	6439	89.5	95.0	Do.
	35 750	.....	.....	1864	5.2	31 088	86.9	92.8	

HEAT NO. 1x3632—Continued

6	7340	7234	505	694	8.2	6067	82.6	88.3	Aug. 18, 1915
7	7200	7099	505	710	9.8	5956	84.1	88.2	Do.
8	7260	7158	163	351	4.8	6400	88.1	93.5	Do.
9	7240	7135	336	561	7.7	6146	84.8	90.4	Do.
	29 040	.....	.....	2316	8.0	24 669	85.0	90.2	

HEAT NO. 2x4510—Continued

11	7510	7400	1515	2179	29.0	4717	62.8	67.4	Aug. 19, 1915
12	7340	7229	336	624	8.5	6112	83.2	89.2	Do.
13	7190	7088	2356	2846	39.5	3873	53.8	56.7	Do.
14	7320	7209	1682	2061	28.2	4694	64.1	68.3	Do.
15	7480	7373	1815	2051	27.4	4825	64.5	69.1	Do.
	36 840	.....	.....	9761	26.5	24 221	65.8	70.2	
Total	101 630	100 131	10 054	13 941	13.7	79 978	78.7	83.8	

HEAT NO. 1x3632—Continued

10	.....	7180	.....	.....	.....	.....	.....	.....	Aug. 18, 1915

BLOOMING MILL.—All ingots were rolled in 13 passes in a two-high mill to blooms 8 by 8⅛ inches. The English made ingots entered the blooming mill with the sink head toward the rolls. One half received the first, or "squaring up," pass in this direction, no draft being given on second pass; the other half were given no draft on the first pass, the "squaring up" being done on pass No. 2 (Table 10). The comparison ingots were rolled according to ordinary practice, entering the rolls the small end first.

2. BLOOMS

Each ingot was cut into 2 blooms (8 by 8⅛ inches) as shown in Figs. 1 and 2, and rolled directly into rails, that is, without reheating of blooms. The second bloom from sink-head ingots made 2 rails (C and D), and that from the comparison ingots made

3 rails (C, D, and E). The first bloom from the sink-head type of ingot made a B and, generally, a short A rail; the first bloom from the comparison type of ingot made a B, A, and (short) X rail. The blooms were sheared top and bottom as little as possible for the safe passage of the bar through the rolls. Each piece cut was marked, weighed, and preserved.

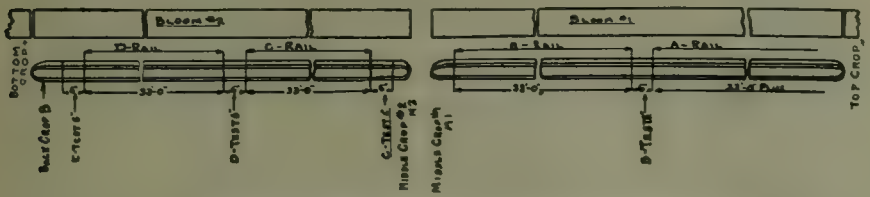


FIG. 1.—*Method of cutting blooms and rails from sink-head ingots*

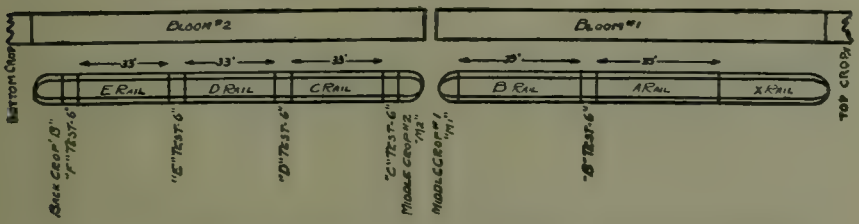


FIG. 2.—*Method of cutting blooms and rails from comparison ingots*

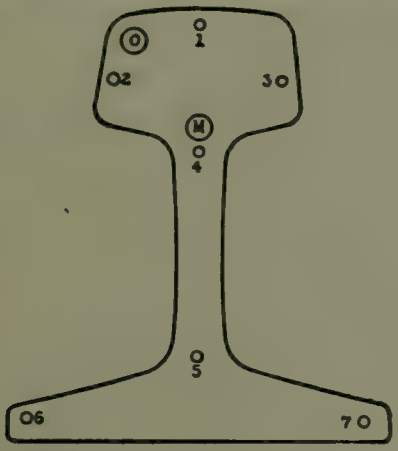


FIG. 3.—*Section of 100-pound P. S. rail, showing location of various* tests

Drillings for chemical analysis were taken at "O" and "M." Hardness determinations were made at the other positions indicated

3. RAILS

In the rail mill there are 6 passes in the roughing rolls, 4 in the intermediate rolls, and 1 finishing pass, making a total of 24 passes

from ingot to finished rail. The rails, which were Pennsylvania 100-pound section (Fig. 3), were cut at the hot saw as shown in Figs. 1 and 2.

The temperature of all rails was taken at the finishing pass. The rails were inspected for surface defects, piping, and discard to physically sound steel. The classification into "firsts" and "seconds" from surface conditions was made by the Pennsylvania Railroad Co. inspectors (Table 12); the discard to "physically sound steel" was made by cutting back in 5-foot lengths.

TABLE 12.—Classification of Rails Rolled from Sink-Head and Comparison Ingots [a]

Rails from sink-head ingots				Rails from comparison ingots			
Ingot number	B rail	C rail	D rail	B rail	C rail	D rail	E rail
1	1	1	1	1	1	1	1
2	1	1	1	1	1	1	1
3	1	1	1	1	1	1	1
4	2	2	2	1	1	1	1
5	(a)	2	2	1	1	1	2
6	1	2	2	1	1	1	1
7	2	2	2	1	1	1	1
8	2	1	2	1	1	1	1
9	1	1	1	1	1	1	1
10	2	1	1	(b)	1	1	1
11	2	2	2	1	1	1	2
12	2	1	2	1	1	1	1
13	2	2	1	(b)	2	1	1
14	1	1	1	(b)	1	1	1
15	2	2	2	1	1	1	1
16	2	2	2				
17	1	1	1				Per ct.
18	1	1	1	No. 1 rails		53	93
19	1	1	1	No. 2 rails		4	7
20	2	2	2	Total		57	100
21	1	2	1				
22	1	1	1				
23	1	1	2				
24	1	1	1				
25	2	2	2				
26	1	1	1				
27	2	2	2				
28	2	2	2				
29	1	2	1				
30	2	2	2				
31	1	2	1				
32	2	1	2				
33	1	1	2				
34	2	2	2				
35	1	1	1				

		Per ct.
No. 1 rails	43	41
No. 2 rails	61	59
Total	104	100

[a] Inspection made by Pennsylvania Railroad Co. inspectors.
[b] Sent to Bureau of Standards.

The HA, the MX, and MA rails were held for tests to be described later. All pieces of rail were stamped to identify the ends.

4. TESTS

MECHANICAL TESTS.—The first sound 5-foot length obtained was taken as the drop-test piece. The test was made in the usual way by dropping a 2000-pound tup from a height of 18 feet upon the head of the rail which was supported at 2 points 3 feet apart. This test gives record of permanent set, elongation of the base, and the number of blows required for the destruction of the rail.

The 6-inch test pieces (B, C, D, etc., Figs. 1 and 2), the drop-test pieces, and the specimens from the MA position were examined for ultimate tensile strength, yield point, elongation (2-inch gage length), reduction of area, and hardness by Brinell and scleroscope. All specimens for the tension tests were cut longitudinally.

METALLOGRAPHIC TESTS.—There were taken of each test-piece section, a sulphur print, a light etching, and two photomicrographs at 100 diameters. Photomicrograph A was taken at upper part of web, B corresponds approximately with O position (Figs. 8 to 12, inclusive). These tests were also made on sections of the 5-foot pieces of the MX, MA, and HA rails to accompany the determinations of discard for chemical segregation.

CHEMICAL TESTS.—Determinations were made of the amounts of carbon, sulphur, phosphorus, manganese, and silicon at the "O" and "M" positions (Fig. 3) for test pieces (B, C, D, etc.) of all the rails; samples from the 5-foot pieces of MX, MA, and HA were also analyzed. Some specifications contain a clause which provides for the rejection of rails when the M position shows a carbon content more than 12 per cent greater (positive segregation) or more than 12 per cent less (negative segregation) than the O position. In the present investigation, all material showing more than 12 per cent positive or negative carbon segregation was considered as unavailable for rails. The railroad using this specification has found from experience that 12 per cent is the maximum safe limit of segregation.

III. DESCRIPTION OF OPERATIONS AND DISCUSSION OF RESULTS

1. INGOT PRACTICE

Since the sink-head ingots were cast in England and rolled in this country, they were necessarily subjected to the unusual process, at least in rail manufacture as ordinarily practiced, of reheating from the cold condition. The plan was considered of

allowing at least some of the comparison ingots to go "stone cold" and reheat them before rolling. However, in view of the fact that the ordinary type of ingot is not designed to meet this condition, which practically never obtains in practice and which, therefore, would be a possible misleading condition to impose on the comparison ingots and of no practical value, the comparison ingots were all rolled directly after being cast. These ingots were stripped from 20 to 30 minutes after casting and put into the soaking pits while still hot, where they remained from 1 hour and 40 minutes to 2 hours and 23 minutes.

The sink-head ingots after preliminary heating were charged into relatively cool pits (for temperature details see Section III, 2, Temperature Observations). The heat was gradually turned on after ½ hour and the ingots remained in the pits from 17⅓ hours to 21⅓ hours. The 35 sink-head ingots which were rolled were divided into three series and rolled on successive days, August 16, 17, 18, 1915. Three heats of comparison ingots were rolled, 1 on August 16 and 2 on August 18. There was no delay or other unusual condition in the mill during the rolling. The ingots of a group were rolled in succession, without intermission, and after the mill had been warmed up by rolling at least one complete heat of the regular product of the mill.

2. TEMPERATURE OBSERVATIONS

Temperature observations were taken of the soaking pits before, and at intervals, after immersion of ingots of both types; of all ingots in the blooming mill; and of rails from both series of ingots at the finishing pass. In order to heat them gradually, the Hadfield ingots lay on the surface of the soaking pits approximately 12 hours before charging. They were too hot to bear the hand comfortably when charged, at which time their estimated temperature was about 50° C (122° F). The pits which received these sink-head ingots were allowed to cool to the temperature of 600° C (1112° F)—750° C (1382° F) (Table 7). The immersion of the ingots further cooled the pits by some 200° C (392° F) as measured immediately after charging. The gas was turned on the pits ½ hour after charging. The comparison ingots were charged into the soaking pits hot, according to the usual practice.

Table 7 shows the values of the pit temperatures (1) before charging, (2) about 14½ hours after charging, and (3) just before drawing the sink-head ingots. These temperatures were taken with a platinum-rhodium thermocouple connected to a pivot

type of indicator. The couple was inclosed in nickel sheath except the tip, which was exposed. In some cases the temperatures were obtained by the use of an optical pyrometer of the Morse type.

By means of the optical pyrometer temperatures of the ingots were taken during the rolling. Average values obtained from 7 or 8 observations with optical pyrometer at alternate passes, after the first roughing pass, were taken of the ingots in the blooming mill. The temperature of the steel at the surface was lowered about 17° C (63° F) to 40° C (104° F) during the rolling into blooms. Typical illustrations of rolling temperatures are given in Table 9 for several ingots of both types. Table 7 contains finishing temperatures of Hadfield rail bars taken at the finishing pass by sighting on the center of base of rail bar with an optical pyrometer. Each observation is the average of two readings.

In Table 8 are similar data for the comparison ingots and rails, except that the pit temperatures were not taken. It will be noted that good uniformity of rolling and finishing temperatures was obtained.

A series of measurements was taken to compare the temperatures of this investigation taken at the finishing pass with those taken [3] at the hot saw by sighting on the head of the rail. The reading noted in Tables 7 and 8 of finishing temperatures should be reduced by about 11° C (52° F) to correspond to previous observations at hot saw.

The observed ingot and rail temperatures were taken on the outside oxidized surfaces, which are colder than the interior metal by 75° C (167° F) to 100° (212° F). This would account for the apparent discrepancy between the soaking-pit and ingot temperatures, and would make the average true finishing temperature somewhat higher than 1100° C (2012° F).

3. GENERAL COMPARISON OF THE TWO TYPES OF INGOT

(*a*) Size of Ingots.—The sink-head ingots were of 5200 pounds weight and the comparison ingots of 7300 pounds. In computing the percentage of ingot material available for rails the necessary constant valued deductions—i. e., the bottom-bloom crops and the middle and bottom rail crops—work to the relative disadvantage of the ingot of lesser weight. In was on this account that in Tables 10 and 11 the percentage of material available for

[3] B. S. Tech. Papers, No. 38, Observations on finishing temperatures and properties of rails.

rails is based upon both the original weight of ingot less the weight of the bottom-bloom crops and the initial weight minus the middle and bottom rail crops.

(*b*) Loss in Heating.—The sink-head ingots were weighed while cold before reheating, and the comparison ingots were weighed while hot. The abnormal loss in weight sustained by the former should be deducted from the nominal ingot weight in computing the net ingot. It so happens that the average bottom-bloom crop for the ingot made in an ordinary manner is 0.5 per cent greater than for the sink-head ingot; so that if the loss on heating for the comparison ingots is taken as 0.5 per cent there will be an exact balance. The English-cast ingots remained in the soaking pits some 10 times as long as the comparison ingots. If it is nevertheless preferred to compare actual percentages of material available for rails, that from the sink-head ingots should be increased by 2.5 per cent because there was a loss of 3 per cent, of which some 2.5 per cent can be charged to reheating. When this correction is made it is seen that the average sink-head ingot gives about 84.8 per cent material available for rails instead of 81.9 per cent. Ingot H24 should not be used in the discussion, as it was accidentally given an excessive bloom crop. The expression "material available for rails," as used here and in Tables 10 and 11, refers to only physically sound steel. Subsequent chemical analyses showed a much greater discard was necessary to eliminate segregation above 12 per cent (Tables 17 and 18).

(*c*) Comparison by Heats.—It will be noted that the three heats of comparison ingots have each very distinct characteristics; for example, as expressed in material available for rails, which are the results of varying open-hearth and casting practice. In group M1–M5 the percentage of material available for rails was 86.9 per cent; in group M6–M9, 85 per cent; and in group M11–M15, 65.8 per cent (Table 11). It was for this reason that, in addition to the general average of properties of all Maryland rails, averages were computed for each of the three Maryland heats. The three rollings of the Hadfield ingots show great uniformity, however expressed, and thus indicate that the manufacture of the ingots in all cases apparently was carried out in accordance with a uniform practice.

TABLE 13.—Mechanical and Chemical Characteristics of Rails from Sink-Head Ingots

Rail No.	Average, ADFM,[a] or DFAR [b]	Tension tests				Hardness		Elements determined (per cent)									
		Yield point	Ultimate strength	Elongation, per cent in 2 inches	Reduction of area	Brinell	Scleroscope	Carbon			Manganese		Phosphorus		Silicon		Sulphur	
								O	M	Segregation[c]	O	M	O	M	O	M	O	M
		Lbs. in.	Lbs. in.		Per cent													
HA	Average	62 074	125 986	14.5	23.48	259	33	0.652	0.733	+12.73	0.88	0.85	0.035	0.033	0.137	0.139	0.047	0.050
	ADFM [a]	1 585	2 342	0.7	1.25	6	1	.018	.044	7.84	.04	.03	.003	.002	.008	.007	.003	.005
	DFAR [b]	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HB	Average	63 212	126 381	13.3	23.89	264	31	.649	.664	2.32	.89	.89	.032	.032	.139	.139	.047	.048
	ADFM	1 206	2 027	0.9	1.50	8	1	.016	.018	1.35	.03	.04	.002	.002	.011	.012	.004	.005
	DFAR	+1 138	+395	−1.2	+0.41	+5	−2	−.003	−.069	−10.41	+.01	+.04	−.003	−.001	+.002	0	0	−.002
HC	Average	62 941	126 054	13.6	24.30	260	31	.653	.644	− 1.27	.89	.88	.032	.031	.139	.138	.047	.046
	ADFM	1 109	2 130	0.7	1.18	9	1	.017	.017	1.68	.03	.04	.002	.002	.011	.012	.003	.003
	DFAR	867	+68	−0.9	+0.82	+1	−2	+.001	−.089	−14.00	+.01	+.03	−.003	−.002	+.002	−.001	0	−.004
HD	Average	63 029	126 158	13.6	24.47	260	30	.650	.621	4.48	.89	.88	.032	.031	.139	.137	.047	.044
	ADFM	1 310	2 381	0.7	1.14	7	1	.017	.022	1.81	.03	.03	.002	.002	.011	.012	.004	.004
	DFAR	+955	+172	−0.9	+0.99	+1	−3	−.002	−.112	−17.21	+.01	+.01	−.003	−.002	+.002	−.002	0	−.006
HE	Average	62 264	124 303	14.2	25.70	256	30	.637	.639	+ 0.40	.89	.88	.032	.037	.137	.137	.046	.046
	ADFM	1 418	2 900	0.6	1.63	10	1	.019	.023	1.42	.03	.04	.002	.003	.011	.011	.004	.004
	DFAR	+190	−1 683	−0.3	+2.22	−3	−3	.015	.094	12.33	+.01	+.03	−.003	−.001	0	−.002	−.001	−.004
GENERAL AVERAGES																		
H	General averages	62 704	125 771	13.8	24.38	260	31	0.648	0.680	1.94	0.89	0.88	0.032	0.033	0.138	0.138	0.047	0.047
	ADFM	1 326	2 356	0.8	1.34	8	1	.017	.025	2.75	.03	.04	.002	.002	.010	.011	.004	.005

[a] Average deviation from mean.
[b] Deviation from A rail.
[c] Shows percentage carbon variation of M from O position.

TABLE 14.—Mechanical and Chemical Characteristics of Rails from Comparison Ingots

Rail No.	Average, ADFM[a] or DFAR[b]	Tension tests				Hardness		Carbon		
		Yield point	Ultimate strength	Elongation, per cent in 2 inches	Reduction of area	Brinell	Sclero-scope	O	M	Seg.[c]
		Lbs./in.²	Lbs./in.²		Per cent					
M1-5A	Average	60 721	120 960	13.5	23.16	246	32	0.601	0.736	+22.32
	ADFM[a]	821	2 928	1.0	3.23	5	1	.023	.056	6.65
M6-9A	Average	60 115	123 600	12.8	21.42	252	33	.652	.730	+11.88
	ADFM	885	1 075	1.0	1.61	8	0	.009	.045	6.04
M11-15A	Average	64 963	129 695	13.3	23.43	267	34	.682	.738	+ 8.52
	ADFM	1 322	1 226	0.2	1.50	7	1	.150	.058	9.60
M1-5B	Average	64 145	127 910	11.6	20.80	260	33	.641	.709	+10.60
	ADFM	1 048	2 048	0.5	1.61	3	1	.011	.041	4.78
M6-9B	Average	63 140	128 188	11.6	20.18	259	33	.675	.715	+ 6.18
	ADFM	238	938	0.6	1.20	6	1	.008	.095	1.51
M11-15B	Average	64 484	130 140	12.5	23.92	263	33	.661	.671	− 1.53
	ADFM	345	888	0.4	0.70	1	1	.070	.190	1.49
M1-5C	Average	61 752	124 290	12.5	24.18	244	32	.634	.613	− 3.40
	ADFM	411	548	0.4	0.57	7	1	.007	.030	4.09
M6-10C	Average	61 982	127 520	12.0	20.81	255	34	.673	.673	0.00
	ADFM	593	556	0.6	0.56	2	1	.011	.021	2.12
M11-15C	Average	62 415	126 850	13.1	26.77	354	32	.673	.646	− 4.02
	ADFM	704	1 720	0.5	0.71	3	1	.070	.050	0.89
M1-5D	Average	61 254	123 295	13.1	26.70	245	32	.635	.596	− 6.12
	ADFM	1 207	1 016	0.7	1.40	5	1	.011	.016	1.04
M6-10D	Average	61 158	125 700	12.5	23.75	246	31	.671	.635	− 5.43
	ADFM	686	940	0.4	0.50	2	0	.003	.009	1.56
M11-15D	Average	62 290	126 750	13.6	26.95	252	31	.674	.607	− 9.88
	ADFM	772	1 060	0.3	0.26	4	1	.080	.070	1.58
M1-5E	Average	59 233	120 465	14.7	30.40	240	31	.623	.534	−14.35
	ADFM	1 150	722	0.6	1.30	3	0	.003	.010	1.32
M6-10E	Average	59 000	123 055	13.9	26.72	246	33	.650	.617	− 4.99
	ADFM	446	874	0.4	1.44	2	1	.008	.013	2.15
M11-15E	Average	60 944	124 050	14.3	29.26	242	31	.643	.570	−11.40
	ADFM	1 372	1 020	0.4	0.55	6	1	.110	.110	1.25

M1-5F	Average	67 592	117 190	15.5	32.34	234	32	.585	.585	+ 1.73
	ADFM	500	1 048	0.6	0.59	8	1	.004	.043	6.07
M6-10F	Average	59 395	121 735	15.2	30.03	244	32	.637	.643	− 0.53
	ADFM	382	1 248	0.5	0.73	5	2	.007	.008	2.05
M11-15F	Average	59 450	120 065	15.8	32.68	249	32	.613	.611	− 0.39
	ADFM	1 028	1 502	0.2	0.82	2	0	.040	.080	1.42

SUMMARY BY INGOT POSITION

M-A rails	Average	62 063	124 834	13.2	22.76	255	33	0.644	0.735	+14.41
	ADFM	2 091	3 472	.6	2.14	11	1	.064	.045	7.53
	DFAR	0	0	0	0	0	0	0	0	0
M-B rails	Average	63 979	128 786	11.9	21.73	260	33	.665	.697	+ 5.00
	ADFM	840	1 488	.5	1.19	4	1	.032	.109	2.67
	DFAR	+ 1 916	+3 952	− 1.3	− 1.03	+ 5	0	+ .021	− .038	− 9.41
M-C rails	Average	62 050	126 220	13.5	23.92	251	33	.660	.644	− 2.42
	ADFM	631	1 606	.6	.61	6	1	.029	.034	2.35
	DFAR	− 13	+ 1 388	+ .3	+ 1.16	− 4	0	+ .016	− .091	−11.99
M-D rails	Average	61 567	125 248	13.1	25.80	248	31	.660	.612	− 7.14
	ADFM	999	1 495	.6	.72	4	1	.031	.032	1.39
	DFAR	− 496	+ 414	− .1	+ 3.04	− 7	− 2	+ .016	− .123	− 7.27
M-E rails	Average	59 726	122 523	14.3	28.79	243	32	.638	.573	−10.40
	ADFM	1 068	1 536	.5	1.09	5	1	.043	.044	1.57
	DFAR	− 2 337	− 2 311	+ 1.1	+ 6.03	− 12	− 1	− .006	− .162	− 4.01
M-F rails	Average	58 826	119 663	15.5	31.68	242	32	.611	.616	+ .27
	ADFM	1 052	2 049	.5	.71	8	1	.017	.040	3.18
	DFAR	− 3 237	− 5 179	+ 2.3	+ 8.92	−13	− 1	− .033	− .119	−14.14

SUMMARY BY HEATS

M1-5	Average	60 783	122 352	13.5	26.26	245	32	0.620	0.631	+ 1.80
	ADFM	1 873	3 068	1.2	3.53	9	1	.020	.064	10.84
M6-10	Average	60 757	124 792	13.1	24.03	250	33	.636	.665	+ .98
	ADFM	1 416	2 151	1.2	3.13	6	1	.024	.038	5.64
M11-15	Average	62 431	126 258	13.8	27.17	255	32	.661	.641	− 3.22
	ADFM	1 604	4 681	.7	2.62	3	1	.038	.042	5.14

GENERAL AVERAGE

	General average	61 368	124 545	13.6	25.78	250	32	0.645	0.646	+ 0.16
	ADFM	1 113	1 941	.6	1.01	6	1	.036	.051	3.12

[a] Average deviation from mean. [b] Deviation from A rail. [c] Shows percentage carbon variation of "M" from "O" position.

TABLE 14.—Mechanical and Chemical Characteristics of Rails from Comparison Ingots—Continued

Rail No.	Average, ADFM[a] or DFAR[b]	Manganese		Phosphorus		Silicon		Sulfur		Nickel		Chromium	
		O	M	O	M	O	M	O	M	O	M	O	M
M1-5A	Average	0.64	0.68	0.010	0.013	0.127	0.130	0.058	0.086	0.75	0.75	0.28	0.29
	ADFM[a]	.01	.02	.001	.001	.005	.006	.004	.015	.01	.01	.01	.01
M6-9A	Average	.66	.67	.015	.018	.142	.145	.096	.124	.72	.72	.29	.30
	ADFM	.01	.01	.000	.002	.007	.008	.007	.018	.00	.01	.00	.00
M11-15A	Average	.77	.79	.028	.032	.178	.179	.118	.117	.73	.73	.26	.27
	ADFM	.01	.02	.001	.004	.015	.018	.004	.029	.00	.01	.00	.00
M1-5B	Average	.68	.69	.012	.013	.129	.130	.074	.097	.77	.77	.28	.28
	ADFM	.01	.01	.000	.000	.007	.006	.004	.011	.01	.00	.00	.02
M6-9B	Average	.68	.68	.016	.018	.168	.147	.103	.115	.75	.75	.28	.28
	ADFM	.01	.01	.001	.001	.020	.002	.001	.006	.01	.01	.01	.01
M11-15B	Average	.78	.78	.030	.029	.174	.174	.122	.120	.73	.74	.27	.27
	ADFM	.01	.01	.001	.001	.007	.007	.001	.006	.00	.01	.01	.00
M1-5C	Average	.67	.67	.013	.012	.128	.128	.076	.073	.77	.78	.27	.28
	ADFM	.01	.00	.001	.001	.007	.006	.001	.005	.00	.01	.01	.01
M6-10C	Average	.66	.66	.017	.017	.145	.145	.101	.103	.74	.74	.26	.29
	ADFM	.01	.01	.000	.001	.004	.003	.001	.002	.01	.01	.01	.01
M-11-15C	Average	.78	.78	.029	.027	.173	.172	.118	.114	.74	.74	.27	.27
	ADFM	.01	.02	.000	.001	.008	.008	.003	.002	.00	.00	.01	.00
M1-5D	Average	.67	.66	.012	.016	.127	.145	.007	.094	.78	.74	.29	.29
	ADFM	.01	.01	.001	.001	.008	.001	.003	.004	.00	.01	.01	.01
M6-10D	Average	.67	.67	.016	.012	.144	.127	.100	.069	.74	.78	.29	.28
	ADFM	.00	.00	.001	.001	.002	.008	.002	.003	.00	.01	.01	.01
M11-10D	Average	.78	.78	.028	.027	.173	.172	.119	.108	.74	.74	.27	.26
	ADFM	.01	.00	.001	.001	.009	.006	.002	.003	.00	.01	.01	.01
M1-5E	Average	.67	.67	.012	.011	.130	.130	.073	.060	.77	.77	.28	.28
	ADFM	.01	.01	.001	.000	.006	.006	.001	.001	.00	.00	.01	.01
M6-10E	Average	.66	.66	.016	.016	.148	.147	.097	.090	.74	.75	.29	.28
	ADFM	.01	.00	.001	.001	.001	.001	.003	.003	.01	.00	.01	.00
M11-15E	Average	.78	.78	.028	.024	.175	.176	.114	.096	.74	.73	.27	.26
	ADFM	.01	.01	.001	.001	.010	.007	.001	.002	.00	.00	.00	.01
M1-5F	Average	.67	.65	.010	.011	.132	.133	.065	.070	.77	.77	.28	.28
	ADFM	.01	.02	.000	.001	.006	.006	.001	.005	.01	.01	.01	.01

M6–10F	Average	.66	.68	.016	.018	.147	.151	.096	.097	.73	.74	.29	.28
	ADFM	.00	.03	.001	.003	.002	.006	.003	.003	.01	.01	.01	.01
M11–15F	Average	.78	.77	.026	.026	.177	.178	.104	.106	.74	.73	.26	.26
	ADFM	.02	.01	.002	.000	.007	.007	.002	.001	.00	.00	.00	.00

SUMMARY BY INGOT POSITION—Continued

M–A rails	Average	0.69	0.71	0.018	0.021	0.149	0.152	0.091	0.106	0.73	0.73	0.28	0.29
	ADFM	.01	.02	.001	.002	.009	.010	.005	.020	0	.01	.00	.00
	DFAR	.00	0	0	.000	0	0	0	0	0	0	0	0
M–B rails	Average	.71	.71	.020	.019	.156	.150	.099	.110	.75	.75	.28	.27
	ADFM	.01	.01	.001	.001	.010	.005	.002	.008	.01	.01	.01	.01
	DFAR	+ .02	0	+.002	+.002	+.007	−.002	+.008	+.004	+ .02	+ .02	.00	− .02
M–C rails	Average	.70	.70	.019	.019	.149	.148	.098	.096	.75	.75	.27	.28
	ADFM	.01	.01	.000	.001	.006	.006	.001	.002	.00	.01	.01	.01
	DFAR	+ .01	− .01	+.001	+.002	.000	−.004	+.007	−.010	+ .02	+ .02	− .01	− .01
M–D rails	Average	.71	.70	.019	.018	.148	.148	.099	.090	.75	.75	.28	.27
	ADFM	.01	.00	.001	.001	.006	.006	.002	.003	.00	.01	.01	.01
	DFAR	+ .02	− .01	+.001	−.003	−.001	−.004	+.008	−.016	+ .02	+ .02	0	− .02
M–E rails	Average	.70	.71	.019	.017	.150	.151	.095	.082	.75	.75	.28	.27
	ADFM	.01	.01	.001	.001	.005	.005	.002	.002	.00	.00	.01	.01
	DFAR	+ .01	.00	+.001	−.004	+.001	−.001	+.004	+.024	+ .02	+ .02	.00	− .02
M–F rails	Average	.70	.70	.017	.018	.152	.154	.088	.091	.74	.75	.28	.27
	ADFM	.01	.02	.001	.001	.005	.009	.002	.003	.01	.01	.01	.01
	DFAR	+ .01	− .01	−.001	−.003	+.003	+.002	−.003	−.015	+ .01	+ .02	.00	− .02

SUMMARY BY HEATS—Continued

M1–5	Average	0.67	0.[illegible]7	0.012	0.012	0.129	0.130	0.071	0.076	0.77	0.77	0.28	0.28
	ADFM	.01	.[illegible]1	.001	.001	.007	.007	.006	.012	.01	.01	.01	.01
M6–10	Average	.66	.65	.016	.017	.146	.147	.098	.103	.74	.74	.29	.29
	ADFM	.01	.02	.001	.001	.003	.004	.004	.010	.01	.02	.01	.01
M11–15	Average	.78	.78	.028	.027	.175	.175	.116	.114	.74	.74	.26	.27
	ADFM	.01	.01	.001	.001	.009	.009	.005	.002	.01	.01	.01	.01

GENERAL AVERAGE—Continued

	General average	0.70	0.71	0.019	0.019	0.151	0.151	0.095	0.096	0.75	0.75	0.28	0.28
	ADFM	.01	.001	.001	.001	.007	.007	.002	.006	.00	.01	.01	.01

[a] Average deviation from mean. [b] Deviation from A rail.

(*d*) INSPECTION OF INGOTS.—The sink-head ingots were examined while cold for surface defects, and the surface condition of all ingots was noted by another observer as they passed through the blooming mill (Tables 4 and 5). There were side splashes, rough sides, and rough corners on nearly all the sink-head ingots, and only 4 of the 35 were noted as clean blooms in rolling, as compared with 8 clean blooms from the 15 comparison ingots.

(*e*) COMPARISON OF CHEMICAL COMPOSITION. (1) *Sink-Head Ingots* (Table 13).—The chemical composition of the rails from the sink-head ingots shows a remarkable degree of uniformity. Thus for the O analysis the average carbon is 0.648 per cent, with an average deviation from mean of 0.017. If we take the C test piece as representing the ingot composition, the average for carbon of C–M is 0.644 per cent and C–O 0.653 per cent, the average deviation from mean in each case being 0.017.

The average phosphorus content of the O position is 0.032 per cent, with an average deviation of 0.002; for sulphur (O position) the average is 0.047 per cent, with a deviation of 0.004. For the M position the concordance is somewhat less good. Manganese and silicon are very uniformly distributed.

(2) *Comparison Ingots* (Table 14).—These ingots were cast with small end uppermost, and, except for a nickel content of 0.75 per cent and chromium content of 0.28 per cent, were of a composition and uniformity comparable with the sink-head type, the average O position having a carbon content of 0.645 per cent (sink head 0.648 per cent), with an average deviation from mean of 0.036. The manganese content is 0.70 per cent, as compared with 0.89 per cent for the sink-head ingots; the phosphorus content is 0.019 per cent, while in the sink-head ingot it is 0.047 per cent. Group M11–M15 has an average phosphorus content of 0.028 per cent, which quite closely agrees with that of English-cast ingots. This subgroup is also from its method of manufacture the best to compare with the imported ingots.

The sulphur content of the comparison ingots follows the grouping by heats; the O analysis of C rail, for example, for M1–M5 has a sulphur content of 0.076 per cent; for M6–M10 the sulphur is 0.101 per cent; for M11–M15 the sulphur is 0.118 per cent. The manganese and silicon of the first two groups are practically identical, while both are slightly higher in the third group (M11–M15). The chromium and nickel contents are practically constant at 0.28 per cent for the former and 0.75 per cent for the latter for all three groups.

4. THE SINK-HEAD INGOTS, THEIR MANUFACTURE AND CLASSIFICATION

There were 37 ingots of this Hadfield type cast large end up and each provided with a sink head (Table 1 and Fig. 4). The ingots were fed by the Hadfield method in the usual manner, with charcoal and blast continued for about 20 to 40 minutes until the molten steel had set on the top of the head.

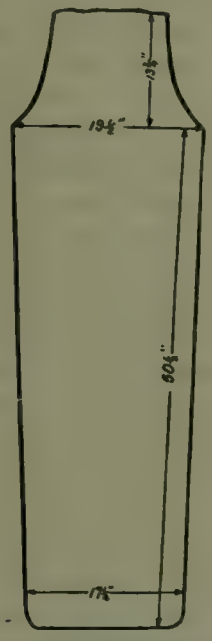


FIG. 4.—*Sink-head ingot*

The steel was made by the converter process, a small furnace being used, and each ingot represents a heat. These ingots are of remarkable uniformity of chemical composition, weight, and sink-head characteristics (Table 2). About 30 to 40 seconds were required for pouring each ingot.

(*a*) SINK-HEAD EFFECT.—Due to the uncertainty of the location of the sink-head junction, an arbitrary allowance has to be made in advance at hot saw or shears or one risks having the A rails rejected. If the total top discard (bloom and rail) is adjusted to 13 per cent there will be no rails rejected on account of sink head; if this is set at 8 per cent one would expect about one-half the A rails to contain surface imperfections caused by the sink head and ingot junction (Table 10). The sink head alone entails on the average a discard of about 9.1 per cent of the Hadfield ingot.

The ingot usually enters the rolls with large end and sink head first; it appears, however, to make no difference which end of ingot is first subjected to the "squaring-up" pass. The presence of the sink head appears to present no difficulties in either the blooming mill or rail mill. One ingot cracked at the bottom of the sink head during rolling, but this was without effect on the passage of this bloom through the rolls.

(*b*) Piping in Sink-Head Ingots.—Two of the 35 sink-head ingots were examined as rails had pipes, detected in the mill, extending into the ingot 10 or 11 per cent, or to some 5 per cent below the sink head in some cases (Table 10). All piping would appear to be eliminated with a discard of 11 per cent, which is very nearly that required to eliminate all sink-head imperfections and carbon segregation above 12 per cent.

(*c*) Influence of Surface Condition of Ingots on Rail Classification.—It was possible to make a very careful examination for flaws in the surfaces of the sink-head ingots while cold; their condition during rolling, as well as of the comparison ingots, was also noted. In this way some idea of the relation of "second" rails to ingot surface may be established (Tables 4, 5, and 12).

There were a relatively large number of "second" rails from the sink-head ingots, 61 out of a total of 104 rails, or 59 per cent. There were only 4 in 57 from the comparison ingots, or 7 per cent. The A rails were not included for either group. This inspection was made by Pennsylvania Railroad Co. inspectors.

There appears to be no reason why ingots of the sink-head type should not be as well surfaced as any other, and the high percentage of seconds should not be attributed to the type of ingot, as the bottom of the sink head was well within the A rail, which was not classified. Probably the severe and unusual reheating conditions, together with the original surface as influenced by condition of the molds and teeming practice, contributed somewhat toward producing the large number of "seconds" from the sink-head ingots. A large percentage of "seconds" was similarly obtained from the reheated Hadfield ingots reported on by Hadfield and Burgess.[4]

5. THE COMPARISON INGOTS, THEIR MANUFACTURE AND CLASSIFICATION

There were 15 Maryland ingots, in 3 groups of 5 each, of about 7300 pounds each, top-poured into ingot molds with small end

[4] See footnote 1.

up. (See Table 1 for dimensions.) They were rolled as portions of three heats on different dates, the total number of ingots in a heat being 13 or 20. The time of teeming into molds was about 35 seconds each. The product from each heat thus forms a distinctive group and should be considered separately. The ingots were selected arbitrarily before being cast as the five to be poured in succession after the second one of the heat. The heats were three of a series of rail steel from the ordinary output of the open hearth and were not especially chosen.

The first 10 ingots, heats 2×4497 (M1–M5) and 1×3632 (M6–M10) were apparently made from rising steel, while ingots M11–M15 of heat 2×4510 were from quiet or "killed" steel; ingots M6–M15 were deoxidized with aluminum; ingots M1–M10 were chilled on top, M1–M5 with cast-iron caps and M6–M10 with water.

The effects of these differences in furnace and casting practice are very striking on the finished product. Thus the deoxidized flat-top ingots (M11–M15) show, as would be expected, pronounced piping; for this group the average discard to physically sound steel is 26.5 per cent and ranging from 8.5 to 39.5 per cent. With this discard may be compared the discard of 5.2 per cent for the first Maryland group and 8 per cent for the second, and except in 2 cases out of 35 a negligible discard for piping below the sink head of the Hadfield ingots. The small discard (6.6 per cent) to sound steel for the average of the first two Maryland groups is, however, misleading, as there may exist internal or inclosed pipes, especially for the second or deoxidized group. In the first comparison group (M1–M5) one would expect to find a spongy, segregated structure in the upper part of the ingot. This is brought out in some of the sulphur prints of rail from the upper part of the ingots of this group, a typical one being shown in Fig. 8.

Of the first group, M5 contains a very pronounced pipe near the bottom of the ingot in the F position (Fig. 9). The question may be raised as to whether the practice of chilling the ingot top does not tend to produce internal pipes which may be distributed anywhere along the center line of the ingot.

The phenomenon of segregation, which is intimately allied to the furnace and casting practice for the comparison ingot, is discussed separately. The mechanical properties, as brought out by the several tests—ultimate strength, yield point, elongation,

reduction of area, hardness and drop test—show less dependence on the processes of manufacture.

6. COMPARISON OF CHEMICAL SEGREGATION OF INGOTS OF BOTH TYPES

Each comparison ingot furnished 6 and each sink-head ingot 4 rails. In comparing the segregation of the A test piece it should be remembered that for the sink-head ingots this position is that of the drop-test piece at about 9 per cent below the top (including sink head), while for the comparison ingots the A test piece is at the junction of the X and A rails, or at 10.6 per cent from the top of ingot. Therefore they are in sufficiently similar positions for comparison, except that 10.6 per cent of a comparison ingot (7260 pounds) represents 770 pounds of steel, or 23 feet of rail, while 9.6 per cent of a sink-head ingot (5300 pounds) represents 509 pounds, or only 15 feet of rail, or the A position in the comparison has a rail discard 8 feet greater than the sink-head ingot on the average.

The E position of the rails from sink-head ingots should be compared with the position of the comparison ingots F, the sink head D with comparison E, the sink head C with comparison C and D, and the sink head B with comparison B.

For both types of ingot the longitudinal segregation of carbon is very slight along the O positions, while for the M positions, or along the center line of the ingot, it is quite marked, the carbon being greatest at A near the top of the ingot, decreasing to the H–D (or M–E) position, and rising again as the bottom of the ingot is approached. The results given are for the three types of comparison ingot, but for this effect, the type of ingot appears to be less important than for other factors.

The segregation of the sink-head ingots and of the three groups of ingots made with small end uppermost are discussed separately with reference to influence and method of manufacture.

(*a*) Segregation in Sink-Head Ingots.—The sink-head ingots were of "piping" steel deoxidized with aluminum. Each represents a separate heat of converter steel.

These ingots were in general of remarkable uniformity as regards segregation (Table 15), thus showing the furnace and casting practice also to have been very uniform. At the A test piece, 9.1 per cent below the top of the ingot on the average, the segregation, though variable, is always positive; that is, the carbon content of the M position is greater than that of the O position. Fourteen of

35 ingots have a segregation greater than 12 per cent and 3 greater than 25 per cent; for one of these (H13), the A position is only 5.1 per cent below the top of the ingot (Table 10).

For these 35 sink-head ingots the discard for segregation above 12 per cent is located 13 per cent (plus or minus 1.86) below top of ingot, with a maximum of 17.5 per cent and a minimum of 5.1 per cent (Tables 10 and 17).

TABLE 15.—Percentage Carbon Segregation in Rails from Sink-Head Ingots

Ingot	Rail A	Rail B	Rail C	Rail D	Rail E	Ingot	Rail A	Rail B	Rail C	Rail D	Rail E
H1	13.02	−0.15	−3.75	−4.59	1.23	H21	11.91	1.66	−0.75	0.45	2.61
H2	9.44	.00	−2.67	−6.77	−1.60	H22	2.67	− .75	.31	−3.48	− .30
H3	11.65	−5.33	−3.89	−7.42	− .79	H23	24.35	.77	−1.69	−4.09	−1.42
H4	16.49	4.83	−3.82	−3.41	− .62	H24	21.94	−2.26	−2.88	−2.93	−4.55
H5	.....	.....	−1.29	−1.30	− .44	H25	5.17	2.68	1.52	−2.56	2.73
H6	9.25	4.86	−1.73	−6.40	2.78	H26	34.16	.60	−1.22	−7.11	.92
H7	19.79	.30	−1.95	− .15	− .60	H27	4.76	1.38	.00	−4.48	3.09
H8	3.34	1.68	− .15	−5.20	1.57	H28	26.77	2.97	−3.12	−5.43	.16
H9	23.77	4.87	−1.12	−6.20	− .34	H29	10.50	3.58	−1.72	−7.55	.49
H10	[illegible]	2.17	.31	−4.54	3.09	H30	15.57	2.54	− .47	−4.32	− .97
H11	4.69	3.74	.32	−5.16	.32	H31	12.89	2.95	.31	−3.70	.62
H12	2.16	1.44	−2.41	−6.52	− .57	H33	3.83	2.06	−7.41	−2.68	.60
H13	46.69	−3.11	−4.35	−1.27	−2.79	H34	10.11	6.75	−2.72	−5.16	−2.96
H14	2.48	1.49	.00	−4.32	.30	H35	10.33	3.32	−5.11	−6.16	1.46
H15	7.83	4.08	−4.63	1.18	1.63	H36	15.48	2.37	2.54	−4.59	− .96
H16	13.93	4.52	−2.33	−9.74	−1.41	Average	+12.73	+2.32	−1.27	−4.48	+0.40
H17	1.50	4.75	1.22	−5.33	3.29	ADFM[a]	7.48	1.35	1.68	1.81	1.42
H18	5.20	1.96	− .30	−3.46	2.14						
H19	9.66	2.85	.00	−7.28	.81						
H20	1.04	2.60	−1.96	−5.07	1.73						

[a] Average deviation from mean.

TABLE 16.—Percentages Carbon Segregation in Rails from Comparison Ingots

Ingot	Rail A	Rail B	Rail C	Rail D	Rail E	Rail F
M1	[illegible]	7.17	−8.80	− 5.07	−12.34	−13.45
M2	30.39	27.55	.31	− 7.73	−13.06	3.93
M3	13.30	9.63	.96	− 5.79	−15.20	4.44
M4	20.17	5.28	−8.23	− 4.41	−15.89	2.96
M5	16.85	3.37	−1.25	− 7.61	−15.24	10.75
M6	16.51	9.20	−4.98	− 5.93	− 3.71	.62
M7	19.34	5.07	3.68	− 3.44	− 8.79	.00
M8	2.51	5.05	.91	− 5.76	− 6.65	.64
M9	9.15	5.38	.73	− 8.27	− 1.42	− 5.91
M10	.....	.....	− .31	− 3.73	− 4.41	.....
M11	3.08	− .87	−4.95	− 8.69	−11.78	− 1.16
M12	32.52	−1.61	−3.61	−11.00	−11.68	− 2.08
M13	.....	−1.98	−5.31	− 7.12	−13.82	− 1.47
M14	4.74	−6.26	−2.48	−11.92	− 8.62	.16
M15	.15	3.07	−3.73	−10.66	−11.09	2.61
Average	+14.41	+5.00	−2.42	−7.14	−10.40	+ .27
ADFM[a]	7.53	2.67	2.35	1.39	1.57	3.18

[a] Average deviation from mean.

At A the average segregation is +12.73 per cent, ±7.48; at B the segregation is +2.32, ±1.35, with only 4 negative values; at

C it is -1.27 per cent, ± 1.66, with 10 positive values; at D it is 4.48 per cent, ± 1.81, with only 2 positive values; and at E only $+0.40$ per cent, ± 1.42, with 15 negative values (Table 15).

(*b*) SEGREGATION IN COMPARISON INGOTS. (1) *Group M1–M5 (Heat 2×4497).*—This heat has no aluminum addition in molds, and cast-iron caps were put on the tops of the ingots as soon as poured. It was found that the ingot tops were rounded after the steel had solidified; therefore, they were made of rising steel not specially deoxidized in the mold. The ingots were stripped 20 minutes after casting and remained in soaking pits from 2 hours and 10 minutes to 2 hours and 23 minutes. This is about as rapid handling of ingots in the molds as it is possible to have, although the time in pits could have been lessened. The resulting composition and segregation is strikingly characteristic of their history.

TABLE 17.—Necessary Total Discard (Percentage) in Rails from Sink-Head Ingots [a]

[H24 was omitted, as this ingot was given an excessive bottom crop due to error in judgment at mill. *s*=discard for segregation. *p*=discard for physical unsoundness.]

Ingot number	Top	Bottom (*p*)	Middle (*p*)	Chemical	Physical	Total
H1	*s* 16.2	3.0	1.8	16.2	4.8	21.0
H2	*s* 12.3	2.5	1.8	12.3	4.3	16.6
H3	*s* 13.9	3.4	2.6	13.9	6.0	19.9
H4	*s* 17.5	3.4	1.8	17.5	5.2	22.7
H6	*s* 13.4	3.7	1.9	12.4	5.6	18.0
H7	*s* 14.0	3.7	2.1	14.0	5.8	19.8
H8	*s* 12.1	3.9	2.4	12.1	6.3	18.4
H9	*s* 15.0	4.2	2.3	15.0	6.5	21.5
H10	*s* 15.6	3.6	2.0	15.6	5.6	21.2
H11	*s* 14.3	3.7	2.1	14.3	5.8	20.1
H12	*p* 10.7	4.1	2.1	.....	16.9	16.9
H13	*s* 13.0	3.8	1.8	13.0	5.6	18.6
H14	*s* 13.8	3.8	1.9	13.8	5.7	19.5
H15	*s* 12.6	3.3	2.2	12.6	5.5	18.1
H16	*s* 12.4	4.4	1.7	12.4	6.1	18.5
H17	*p* 9.1	4.0	2.4	.....	15.5	15.5
H18	*s* 13.9	3.9	2.4	13.9	6.3	20.2
H19	*s* 11.8	3.6	1.9	11.8	5.5	17.3
H20	*s* 14.0	3.7	2.1	14.0	5.8	19.8
H21	*s* 14.6	3.6	2.3	14.6	5.9	20.5
H22	*s* 11.5	3.4	1.8	11.5	5.2	16.7
H23	*s* 10.6	4.3	2.4	10.6	6.7	17.3
H25	*p* 9.4	4.2	2.3	.....	15.9	15.9
H26	*s* 14.3	3.8	1.9	14.3	5.7	20.0
H27	*s* 9.7	3.2	2.5	9.7	5.7	15.4
H28	*s* 13.8	2.7	2.0	15.8	4.7	8.5
H29	*s* 16.8	3.8	2.0	16.8	5.8	22.6
H30	*p* 9.8	4.3	2.3	.....	16.4	16.4
H31	*p* 8.8	3.0	1.6	.....	13.5	13.5
H33	*s* 12.5	4.6	2.4	12.5	7.0	19.5
H34	*s* 14.4	4.2	1.9	14.4	6.1	20.5
H35	*s* 11.6	4.0	1.8	11.6	5.8	17.4
H36	*s* 15.9	3.9	1.9	15.9	5.8	21.7
Average	13.0	3.7	2.1	11.5	7.2	18.4
Deviation	1.86	.....	.....	.....	.....	.....

[a] The total discard (top, middle, and bottom) includes the discard necessary to eliminate piping and segregation above 12 per cent.

TABLE 18.—Necessary Total Discard (Percentage) in Rails from Comparison Ingots [a]

[*s*=discard for segregation. *p*=discard for physical unsoundness. B, E, D refer to rails having above 12 per cent segregation or having inclosed pipes.]

Ingot number	Top	Bottom	Middle	Chemical	Physical	Total
M1	*s* 31.9	*p* 4.7	*s* 32.0 E+D	63.9	4.7	68.6
M2	*s* 24.8	*p* 4.3	*s* 34.5 B+E+D	59.3	4.3	63.6
M3	*s* 23.9	*p* 3.1	*s* 34.4 E+D	58.3	3.1	61.4
M4	*s* 23.7	*p* 3.7	*s* 34.5 E+D	58.2	3.7	61.9
M5	*s* 28.6	*p* 3.3	*ps* 33.7 E+D	62.3	3.3	66.6
Average	26.6	3.4	33.8	60.4	3.8	64.2
M6	*s* 25.3	*p* 5.0	*p* 1.5	25.3	6.5	31.8
M7	*s* 23.7	*p* 3.9	*p* 1.3	23.7	5.2	28.9
M8	*s* 23.3	*p* 4.0	*p* 1.6	23.3	5.6	28.9
M9	*s* 23.8	*p* 4.8	*p* 1.1	23.8	5.9	29.7
Average	24.0	4.44	1.4	24.0	5.8	29.8
M11	*p* 29.0	*p* 5.1	*p* 1.6	0	35.7	35.7
M12	*s* 19.8	*p* 5.0	*p* 1.7	19.8	6.7	26.5
M13	*p* 39.5	*p* 4.1	[b] *p* 1.0	[redacted]	44.6	44.6
M14	*p* 28.2	*p* 4.6	[b] *p* 1.6	0	34.4	34.4
M15	*p* 27.4	*p* 4.8	[b] *p* 1.9	0	34.1	34.1
Average	26.8	4.7	1.6	4.0	31.1	35.1
Average	26.0	3.8	13.0	30.0	14.1	43.9
Deviation	±3.3	.....	.....	.....	.....	.....

[a] The total discard (top, middle, and bottom) includes the discard necessary to eliminate piping and segregation above 12 per cent.
[b] M13 and M14 contain trace of piping in B position. M15 has a small pipe at F.

The top of the ingot is characterized by high and variable segregation; thus the total top discard required to eliminate segregation above 12 per cent ranged from 23.7 to 31.9 per cent for the five ingots, as compared with a top discard of 2.6 to 9.3 per cent to the physically sound steel; that is, free from pipe (Tables 11 and 18). Within this range of discard for segregation, however, the segregation in a single ingot varied by enormous amounts, such as 26.52 to 94.62 per cent for ingot M2 and −7.36 to 49.28 per cent for ingot M5 (Table 20). This type of ingot is also characterized in all cases, by high negative segregation in the lower portion of the ingot, particularly marked at the E position, where it ranges from −12.34 per cent to −15.89 per cent (Table 16). Except for M1, at the bottom of the ingot F position (−13.45 per cent, Table 16), there is a reversal to relatively small positive segregation. All but M2 contain a well-marked soft streak at F, the bottom of the ingot, and in the case of M5 this streak incloses a pronounced pipe (Figs. 9 and 10). The total material that should be discarded from this type of ingot for segregation ranges from 58.2 to 63.9 per cent (Table 18). Provision should be made for a larger bottom-bloom crop than is customary to eliminate both segregation and piping in the bottom of the ingot. The bottom

rail and the adjacent one from every ingot of this group (M1–M5) should be discarded for segregation. The bottom rail has in addition elements of physical weakness shown by the soft steel streaks which may inclose pipes.

(2) *Group M6–M10 (Heat 1×3632).*—The ingots of this heat had rounded tops on which water was put a few moments after casting; they were also treated while being cast with 2 ounces of aluminum per ton of steel. They were, therefore, of rising steel deoxidized in the molds, and should show somewhat greater tendency to piping than the first group; this is borne out by the results. The ingots of the second group required a discard averaging 8 per cent on account of pipe, while only one of the first group required a discard in excess of this amount (Table 11).

With respect to the B, C, D, E, and F positions for this group, the segregation is relatively low (max., 9.20 per cent, Table 16) and uniform from one ingot to another, as well as within any particular ingot, downward from the B position. The top discard for carbon segregation greater than 12 per cent (in O and M positions) is 23.3 to 25.3 per cent (Table 18), and within this discard the segregation is less extreme than in ingots of the first group, the maximum being about 25 per cent, which obtained in only 2 positions out of 29 (Table 20).

(3) *Group M11–M15 (Heat 2×4510).*—Two ounces of aluminum per ton were added to the ingots M11–M15 for the purpose of deoxidation. The ingot tops, which were flat after pouring into molds, were not chilled with caps or water. These ingots were, therefore, of quiet, piping steel and apparently more nearly approach the type of steel represented by the sink-head ingots than do the other two groups of comparison ingots. They remained somewhat longer in the molds (30 minutes as compared with 20 and 23 minutes) before stripping, and slightly longer in the soaking pits than the other comparison ingots.

The considerable piping at the top of the ingots of this group is very strikingly characteristic of the above open-hearth and casting practice. The carbon segregation in the upper half and at the bottom of this type of ingot is slight, but there is developed negative segregation ranging about 11 per cent at the D and E positions (Table 16). In contrast to the other two types of Maryland ingot the extent of the top discard is determined (Table 18), except for M12, which shows other anomalies, by piping (average discard 26.8 per cent) rather than by segregation (average discard 16.4 per cent, Table 22). The metallographic examination shows

a small pipe in rail M15 at the bottom of the ingot in the F position, and incipient piping in M13 and M14 at the B position.

TABLE 19.—Percentage Segregation of Carbon in the "A" Rails of Sink-Head Ingots [a]

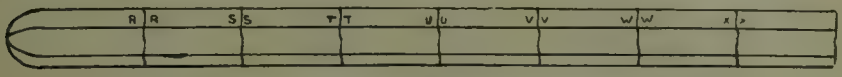


Diagram of cutting up of "A" rail of sink-head ingots

Ingot	Carbon	R	S	T	U	V	W	X
H1	Carbon O	0.632	0.649	0.647	0.650	0.658	0.648	0.647
	Carbon M	.902.	.789	.732	.683	.673	.674	.673
	Segregation	42.72	21.57	13.14	/ 5.08	2.28	4.01	4.02
H2	Carbon O	.610	.636	.635	.632	.634	.637	.636
	Carbon M	.967	.804	.705	.690	.659	.653	.654
	Segregation	58.52	26.42	/ 11.02	9.18	3.94	2.51	2.83
H3	Carbon O	.657	.670	.667	.653	.657	.....	.....
	Carbon M	.746	.708	.674	.682	.673	.....	.....
	Segregation	13.55	/ 5.67	1.05	4.44	2.44	.....	.....
H4	Carbon O	.605	.657	.654	.655	.649	.656	654
	Carbon M	.719	.777	.767	.708	.719	.691	.674
	Segregation	18.84	18.26	17.28	/ 8.09	10.79	5.34	3.06
H6	Carbon O	.640	.641	.641	.643	.644	.....	.....
	Carbon M	.805	.697	.675	.676	.674	.....	.....
	Segregation	25.78	/ 8.74	5.30	5.13	4.66	.....	.....
H7	Carbon O	.630	.669	.647	.667	.663	.662	.561
	Carbon M	.727	.889	.715	.686	.676	.671	.669
	Segregation	15.40	32.88	/ 10.51	2.85	1.96	1.36	1.21
H8	Carbon O	.649	.656	.657	.659	.654	.657	.....
	Carbon M	.758	.690	.673	.661	.662	.664	.....
	Segregation	16.80	/ 5.18	2.44	.30	1.22	1.07	.....
H9	Carbon O	.655	.662	.642	.639	.640	.649	.....
	Carbon M	.804	.742	.686	.658	.662	.674	.....
	Segregation	22.75	12.08	/ 6.85	2.97	3.44	3.85	.....
H10	Carbon O	.661	.638	.638	.643	.653	.643	.....
	Carbon M	.727	.717	.669	.667	.667	.660	.....
	Segregation	9.98	12.38	/ 4.86	3.73	2.14	2.64	.....
H11	Carbon O	.639	.643	.638	.636	.638	.620	.....
	Carbon M	.733	.674	.637	.653	.659	.655	.....
	Segregation	14.71	/ 4.82	.16	2.67	3.29	5.65	.....
H12	Carbon O	.700	.701	.695	.699	.712	.....	.....
	Carbon M	.743	.713	.712	.707	.702	.....	.....
	Segregation	16.14	1.71	2.45	1.14	−1.40	.....	.....
H13	Carbon O	.627	.645	.649	.657	.642	.651	.656
	Carbon M	.786	.739	.664	.667	.658	.666	.663
	Segregation	25.36	14.57	/ 2.31	1.52	2.49	2.30	1.07
H14	Carbon O	.655	.676	.692	.687	.683	.692	.682
	Carbon M	.839	.775	.711	.703	.693	.698	.696
	Segregation	28.09	14.64	/ 2.75	2.33	1.46	.87	2.05
H15	Carbon O	.691	.692	.696	.693	.689	.684	.....
	Carbon M	.758	.819	.761	.725	.719	.713	.....
	Segregation	9.70	18.35	/ 9.34	4.62	4.35	4.24	.....
H16	Carbon O	.685	.687	.693	.674	.683	.....	.....
	Carbon M	.776	.731	.689	.706	.715	.....	.....
	Segregation	13.28	/ 6.40	− .58	4.75	4.69	.....	.....
H17	Carbon O	.667	.675	.669	.657	.659	.659	.....
	Carbon M	.699	.705	.686	.688	.670	.687	.....
	Segregation	/ 4.80	4.44	2.54	4.72	1.67	4.25	.....

[a] The rails were broken at about 5-foot intervals for analysis in accordance with diagram to determine the top discard necessary to eliminate carbon segregation above 12 per cent. The point at which the segregation becomes less than this amount is indicated by "/" in the table.

[b] H25 ingot — Carbon O 0.590; Carbon M .702; Segregation 18.98

TABLE 19—Percentage Segregation of Carbon in the "A" Rails of Sink-Head Ingots—Continued

Ingot	Carbon	R	S	T	U	V	W	X
H18	Carbon O	0.672	0.672	0.672	0.662	0.676	.....	.....
	Carbon M	.771	.772	.712	.692	.669	.....	.....
	Segregation	14.73	14.88	/ 5.95	4.53	−1.04	.....	.....
H19	Carbon O	.634	.631	.635	.631	.632	.....	.....
	Carbon M	.732	.701	.655	.654	.656	.....	.....
	Segregation	15.46	/ 11.09	3.15	3.65	3.80	.....	.....
H20	Carbon O	.638	.661	.663	.660	.668	.....	.....
	Carbon M	.752	.686	.689	.684	.679	.....	.....
	Segregation	17.87	/ 3.78	3.92	3.64	1.65	.....	.....
H21	Carbon O	.676	.674	.680	.668	.679	.....	.....
	Carbon M	.809	.707	.708	.692	.697	.....	.....
	Segregation	19.67	/ 4.90	4.12	3.59	2.65	.....	.....
H22	Carbon O	.678	.671	.671	.674	.681	.....	.....
	Carbon M	.725	.680	.682	.688	.707	.....	.....
	Segregation	/ 6.93	1.34	1.64	2.08	3.82	.....	.....
H23	Carbon O	.644	.655	.646	.647	.644	.....	.....
	Carbon M	.790	.703	.673	.669	.665	.....	.....
	Segregation	22.67	/ 7.33	4.18	3.40	3.26	.....	.....
H24	Carbon O	.618	.628	.629	.626	.....	.....	.....
	Carbon M	.835	.791	.698	.657	.....	.....	.....
	Segregation	35.11	25.96	/ 10.97	4.95	.....	.....	.....
H25	Carbon O	.596	.596	.599	.599	.591	.....	.....
	Carbon M	.633	.627	.625	.609	.609	.....	.....
	Segregation	/ 6.21	5.20	4.34	1.67	3.05	.....	.....
H26	Carbon O	.657	.665	.658	.665	.660	0.661	.....
	Carbon M	.858	.760	.714	.685	.678	.689	.....
	Segregation	30.59	14.29	/ 8.51	3.01	2.73	4.24	.....
H27	Carbon O	.655	.666	.654	.661	.650	.643	.....
	Carbon M	.722	.707	.662	.669	.656	.666	.....
	Segergation	/ 10.23	6.16	1.22	1.21	.92	3.58	.....
H28	Carbon O	.642	.645	.657	.647	.649	.647	.....
	Carbon M	.749	.746	.689	.673	.665	.666	.....
	Segregation	16.67	15.66	/ 4.87	4.02	2.47	2.94	.....
H29	Carbon O	.650	.646	.638	.654	.644	.....	.....
	Carbon M	.754	.682	.667	.671	.668	.....	.....
	Segergation	16.00	/ 5.57	4.55	2.60	3.73	.....	.....
H30	Carbon O	.626	.637	.631	.636	.635	.....	.....
	Carbon M	.681	.666	.662	.661	.656	.....	.....
	Segregation	/ 8.79	4.55	4.91	3.93	3.31	.....	.....
H31	Carbon O	.646	.643	.647	.655	.658	.650	.....
	Carbon M	.717	.679	.657	.646	.668	.665	.....
	Segregation	/ 10.99	5.60	1.55	−1.37	1.52	2.31	.....
H33	Carbon O	.776	.675	.679	.672	.678	.....	.....
	Carbon M	.800	.729	.702	.706	.710	.....	.....
	Segregation	18.34	/ 8.00	3.39	5.06	4.72	.....	.....
H34	Carbon O	.659	.655	.661	.665	.656	.....	.....
	Carbon M	.823	.725	.724	.723	.707	.....	.....
	Segregation	24.89	/ 10.69	9.53	8.72	7.77	.....	.....
H35	Carbon O	.622	.633	.639	.639	.625	.633	.....
	Carbon M	.842	.684	.677	.663	.646	.647	.....
	Segregation	35.37	/ 8.06	5.95	3.76	3.36	2.21	.....
H36	Carbon O	.634	.642	.654	.640	.648	.637	.....
	Carbon M	.796	.722	.655	.666	.649	.655	.....
	Segregation	25.55	12.46	/ .15	4.06	.15	2.83	.....

TABLE 20.—Percentage Segregation of Carbon in the "A" and Discard Rails of Comparison Ingots[a]

Ingot	Carbon	L	M	N	O	P	Q	R	S	T	U	V	W
M1	Carbon O	0.488	0.564	0.554	0.573	0.579	0.588	0.616	0.637	0.629	0.638	0.631	0.629
	Carbon M	.619	.630	.731	.848	.808	.791	.750	.740	.707	.683	.717	.662
	Segregation	26.84	11.70	31.95	47.99	39.55	34.52	21.75	16.17	12.40	7.05	13.63	/ 5.25
M2	Carbon O	.511	.558	.602	.622	.630	.644	.619	.641	.....	.....	.....	.....
	Carbon M	.886	1.086	.915	.898	.894	.878	.849	.811	.....	.....	.....	.....
	Segregation	73.39	94.62	51.99	44.37	41.90	36.34	37.16	26.52	/	.....	.....	.....
M3	Carbon O	.600	.625	.635	.665	.663	.616	.667	.676	.673	.....	.....	.....
	Carbon M	.774	.988	.889	.847	.810	.818	.792	.756	.708	.....	.....	.....
	Segregation	29.00	58.08	40.00	27.37	22.17	32.79	18.74	/ 11.83	5.20	.....	.....	.....
M4	Carbon O	.496	.630	.555	.626	.643	.664	.671	.659	.647	.....	.....	.....
	Carbon M	.661	.704	.884	.900	.861	.808	.752	.669	.690	.....	.....	.....
	Segregation	33.27	11.75	59.28	43.77	33.90	21.69	12.07	/ 1.52	6.65	.....	.....	.....
M5	Carbon O	.589	.605	.621	.626	.652	.630	.647	.652	.658	.641	.....	.....
	Carbon M	.672	.777	.927	.919	.844	.786	.761	.700	.734	.721	.....	.....
	Segregation	14.09	28.43	49.28	46.81	29.45	24.76	17.62	7.36	11.55	12.48	/	.....
M6	Carbon O	.618	.648	.662	.666	.677	.674	.684	.683	.690	.684	.687	.....
	Carbon M	.685	.701	.754	.783	.807	.769	.766	.769	.727	.764	.727	.....
	Segregation	10.84	8.18	13.90	17.57	19.20	14.09	11.99	12.59	/ 5.36	11.70	5.82	.....
M7	Carbon O	.640	.651	.662	.654	.666	.679	.678	.685	.689	.686	.677	.....
	Carbon M	.670	.718	.769	.799	.788	.777	.764	.729	.719	.714	.704	.....
	Segregation	4.69	10.29	16.16	22.17	18.32	14.43	12.68	/ 6.42	4.35	4.08	3.99	.....
M8	Carbon O	.585	.666	.655	.657	.662	.667	.667	.697	.691	.691	.687	.....
	Carbon M	.690	.667	.793	.805	.838	.797	.757	.732	.754	.727	.699	.....
	Segregation	17.95	0.15	21.07	22.53	26.59	19.49	13.49	/ 5.02	9.12	5.21	1.75	.....
M9	Carbon O	.626	.654	.671	.656	.700	.688	.699	.705	.706	.706	.703	.....
	Carbon M	.545	.685	.746	.839	.841	.818	.800	.786	.764	.743	.735	.....
	Segregation	−12.94	4.74	11.18	27.90	20.14	18.90	14.45	/ 11.49	8.22	5.24	4.55	.....

[a] The rails were broken at about 5-foot intervals for analysis in accordance with diagram to determine the top discard necessary to eliminate carbon segregation above 12 per cent. The point at which the segregation becomes less than this amount is indicated by "/" in the table.

TABLE 20.—Percentage Segregation of Carbon in the "A" and Discard Rails of Comparison Ingots—Continued

Ingots	Carbon	L	M	N	O	P	Q	R	S	T	U	V	W
M11	Carbon O	0.703	0.723	0.724	0.712	0.701	0.717	0.705	0.688	0.693	0.689	.....	.....
	Carbon M	.770	.799	.770	.728	.758	.708	.698	.661	.669	.701	.....	.....
	Segregation	/ 9.53	10.51	6.35	2.25	8.13	− 1.26	−0.99	−3.92	−3.46	1.74	.....	.....
M12	Carbon O	.620	.671	.670	.701	.710	.699	.698	.692	.696	.680	.....	.....
	Carbon M	.816	.830	.871	.887	.806	.778	.745	.713	.672	.686	.....	.....
	Segregation	31.61	23.70	30.00	26.53	13.52	/ 11.30	6.73	3.03	−3.45	0.88	.....	.....
M13	Carbon O	.697	.694	.711	.712	.716	.713	.720	.707	.724	.709	.....	.....
	Carbon M	.765	.776	.788	.750	.778	.765	.688	.707	.653	.640	.....	.....
	Segregation	/ 9.76	11.82	10.83	5.34	8.66	7.29	−4.44	0.00	−9.81	−9.73	.....	.....
M14	Carbon O	.714	.711	.694	.700	.692	.705	.705	.706	.708	.682	0.701	.....
	Carbon M	.684	.742	.756	.781	.783	.776	.746	.736	.705	.679	.637	.....
	Segregation	−4.20	4.36	8.93	11.57	13.15	/ 10.07	5.82	4.25	−0.42	−0.44	−9.13	.....
M15	Carbon O	.695	.690	.705	.693	.701	.692	.685	.705	.712	.716	.704	0.663
	Carbon M	.664	.816	.797	.770	.815	.780	.722	.690	.696	.671	.677	.706
	Segregation	−4.46	18.26	13.05	11.11	16.26	12.72	/ 5.40	−2.13	−2.25	−6.28	−3.84	6.49

A striking characteristic of this group (M11–M15) is the freedom from segregation in the A position; excluding M12 A for which the segregation is 32.52 per cent, the average for the others is about 2 per cent segregation at the A position (Table 16). M12 also possesses the following irregular segregation characteristics: At A there is segregation to such an extent that the steel is hypereutectoid; at B, a narrow hypereutectoid streak exists: at C there is the same structure as at A; and again at D a heavy hypoeutectoid streak.

(*c*) SEGREGATION OF ELEMENTS OTHER THAN CARBON.—In general, it might be expected that the elements sulphur and phosphorus would follow carbon in segregation characteristics, and that manganese, silicon, chromium, and nickel would show little or no segregation. These expectations are borne out in both the sink-head and comparison ingots.

Longitudinal segregation of phosphorus is absent for the O position and slight for the M position. There is practically no longitudinal or transverse sulphur segregation in the sink-head ingots, while for the comparison ingots the results on sulphur segregation were erratic but on the whole follow that of the carbon. For the elements manganese, nickel, chromium, and silicon there is no well-defined segregation within the ingot.

7. MECHANICAL TESTS

The mechanical tests included the ordinary drop test made on the first 5-foot piece below the top of the ingot showing physically sound steel. For the Hadfield rails this location was, in all but two cases, determined by the junction of sink head with the ingot proper. The 5-foot pieces formed by cutting up the A rails (also the X rail from the comparison ingots), as well as the test pieces, B, C, D, E (and F for the comparison ingots), were examined for hardness (Brinell and scleroscope) and behavior when stressed in tension.

(*a*) METHOD OF EXPRESSING THE RESULTS OF MECHANICAL TESTS.—It was thought best to express measurements, such as yield point, ultimate tensile strength, elongation, etc., in terms of the average and average deviation from the mean.[5]

When using the usual method of expressing results of this kind in terms of maximum and minimum values, undue emphasis is placed upon the occasional widely divergent figure, while the very

[5] The deviation of a single observation is the amount by which that observation departs from the mean or average value. By subtracting each observation from the mean without regard to sign, and averaging these, the average deviation from the mean is obtained.

close agreement that may exist is not brought to light. Graphic plots of the various physical properties of both kinds of steel will be found in Figs. 13 to 17, inclusive.

TABLE 21.—Total Top Discard Required in Sink-Head Ingots[a]

Ingot	Weight of ingot	Bloom crop	Rail crop	Number of 5-foot pieces of rail	Length of cut	Amount of rail to cut	Rail discard, per cent of ingot	Bloom crop and rail discard	Total top discard required, per cent of ingot
	Pounds	Pounds	Feet		Feet	Pounds		Pounds	
H1	5300	190	5	3	20	667	12.6	857	16.2
H2	5300	155	5	2	15	500	9.4	655	12.3
H3	5300	205	11	1	16	533	10.1	738	13.9
H4	5350	270	5	3	20	667	12.5	937	17.5
H5	5300	324	5	1	10	333	6.3	657	12.4
H7	5250	234	5	2	15	500	9.5	734	14.0
H8	5300	142	10	1	15	500	9.4	642	12.1
H9	5300	294	5	2	15	500	9.4	794	15.0
H10	5250	320	5	2	15	500	9.5	820	15.6
H11	5300	325	8	1	13	433	8.2	758	14.3
H12	5350	204	10	0	10	333	6.2	537	10.0
H13	5300	191	5	2	15	500	9.4	691	13.0
H14	5300	231	5	2	15	500	9.4	731	13.8
H15	5300	167	5	2	15	500	9.4	667	12.6
H16	5350	199	9	1	14	467	8.7	666	12.4
H17	5300	145	10	0	10	333	6.3	478	9.0
H18	5300	237	5	2	15	500	9.4	737	13.9
H19	5300	160	9	1	14	467	8.8	627	11.8
H20	5300	276	9	1	14	467	8.8	743	14.0
H21	5300	442	5	1	10	333	6.3	775	14.6
H22	5250	437	5	0	5	167	3.2	604	11.5
H23	5350	134	8	1	13	433	8.1	567	10.6
H24	5300	185	9	2	19	633	11.9	818	15.4
H25	5300	160	10	0	10	333	6.3	493	9.3
H26	5300	160	8	2	18	600	11.3	760	14.3
H27	5300	245	8	0	8	267	5.0	512	9.7
H28	5300	233	5	2	15	500	9.4	733	13.8
H29	5350	566	5	1	10	333	6.2	899	16.8
H30	5300	247	8	0	8	267	5.0	514	9.7
H31	5300	234	7	0	7	233	4.4	467	8.8
H33	5300	228	8	1	13	433	8.2	661	12.5
H34	5300	195	12	1	17	567	10.7	762	14.4
H35	5300	280	5	1	10	333	6.3	613	11.6
H36	5300	342	5	2	15	500	9.4	842	15.9

[a] The above discard includes amount necessary to eliminate piping and segregation above 12 per cent

TABLE 22.—Total Top Discard Required in Comparison Ingots[a]

Ingot	Weight of ingot	Bloom crop	Rail crop	Number of 5-foot pieces of rail	Length of cut	Amount of rail to cut	Rail discard, per cent of ingot	Bloom crop and rail discard	Total top discard required, per cent of ingot
	Pounds	Pounds	Feet		Feet	Pounds		Pounds	
M1	7360	183	5	12	65	2167	29.4	2350	31.9
M2	7030	243	5	8	45	1500	21.3	1743	24.8
M3	7040	185	5	8	45	1500	21.3	1685	23.9
M4	7130	189	5	8	45	1500	21.0	1689	23.7
M5	7190	223	5	10	55	1833	25.5	2056	28.6
M6	7340	189	5	9	50	1667	22.7	1856	25.3
M7	7200	205	5	8	45	1500	20.8	1705	23.7
M8	7260	188	5	8	45	1500	20.7	1688	23.3
M9	7240	225	5	8	45	1500	20.7	1725	23.8
M11	7540	664	5	0	5	167	2.2	831	11.1
M12	7340	288	5	6	35	1167	15.9	1455	19.8
M13	7190	490	5	0	5	167	2.3	657	9.1
M14	7320	379	5	6	35	1167	15.9	1546	21.1
M15	7480	236	5	7	40	1333	17.8	1569	21.0

[a] The above discard includes amount necessary to eliminate piping and segregation above 12 per cent.

(*b*) ULTIMATE STRENGTH AND YIELD POINT.—The ultimate strength of the steel in rails from both types of ingots are of the same order of magnitude, about 125 000 pounds per square inch.

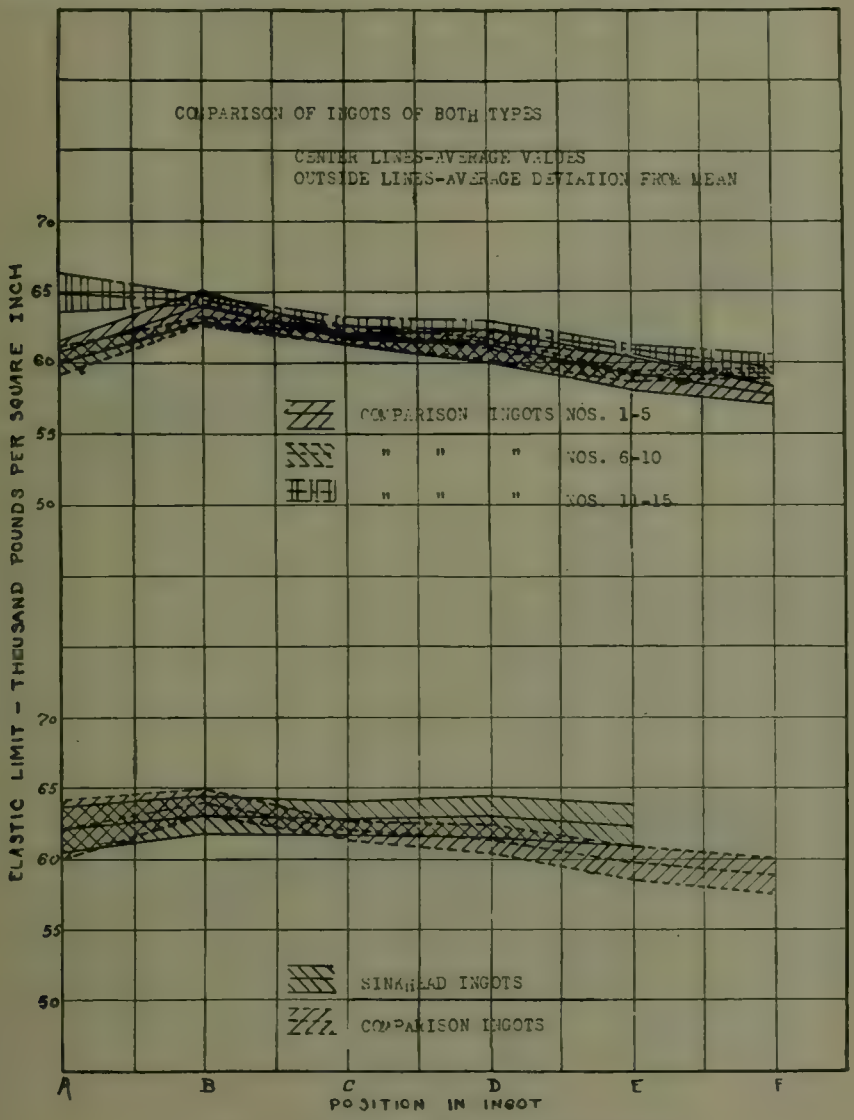


FIG. 13.—*Elastic limit of ingots of both types*

For the 35 sink-head ingots the nonuniformity, expressed as the average deviation from the mean for values at the A, B, C, D, and E positions, is somewhat greater than for the comparison ingots, except for the A position, at which the results are erratic (Tables 13 and 14).

If we consider the ultimate strength throughout the length of the ingot, we note for the sink-head ingots there is practical uniformity down to the E position; for the comparison ingots, on the

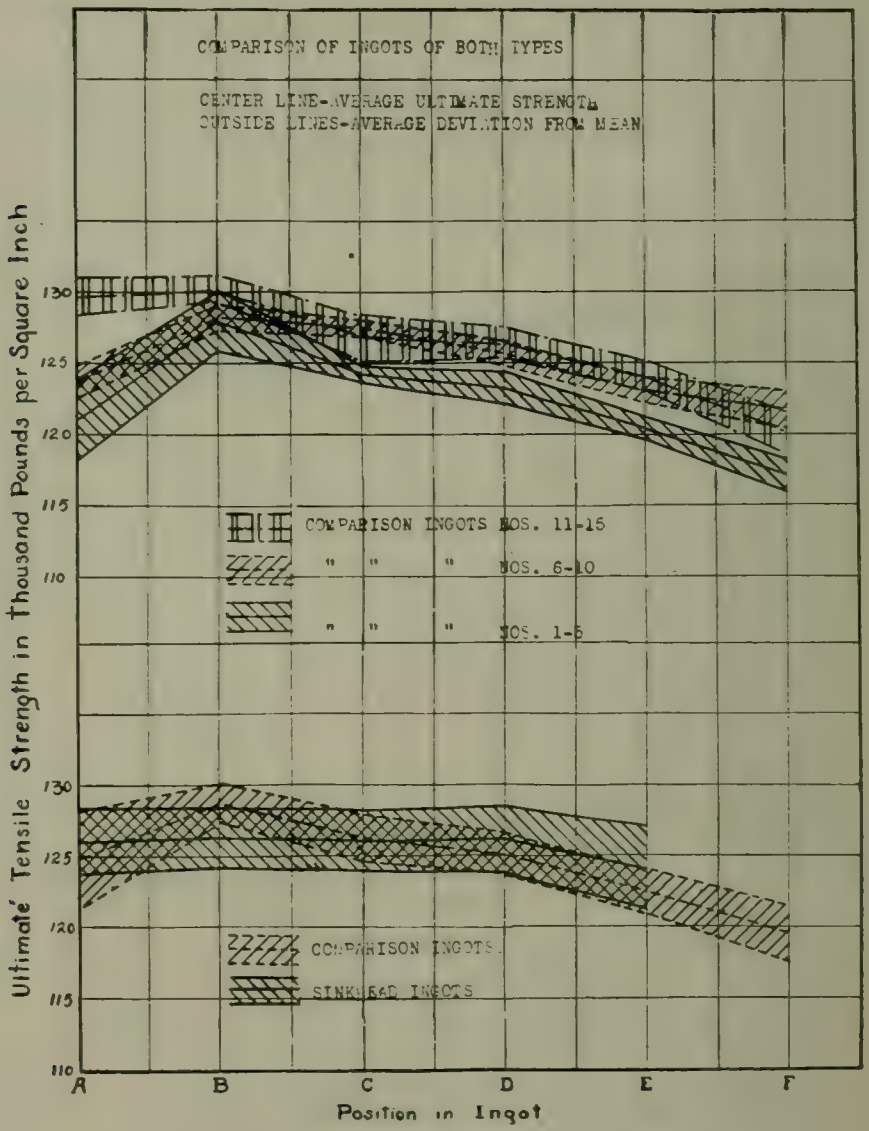


FIG. 14.—*Ultimate strength of ingots of both types*

other hand, there is an abrupt increase in ultimate strength from the A to the B position and a gradual falling off until the bottom of the ingot is reached, so that at the F position the ultimate tensile strength is some 9000 pounds per square inch less than the maximum at B.

If the comparison ingots are considered by groups corresponding to heats, it is seen that the ultimate strength of each group may be distinguished from the others. Thus groups M1–M5 and M6–M10

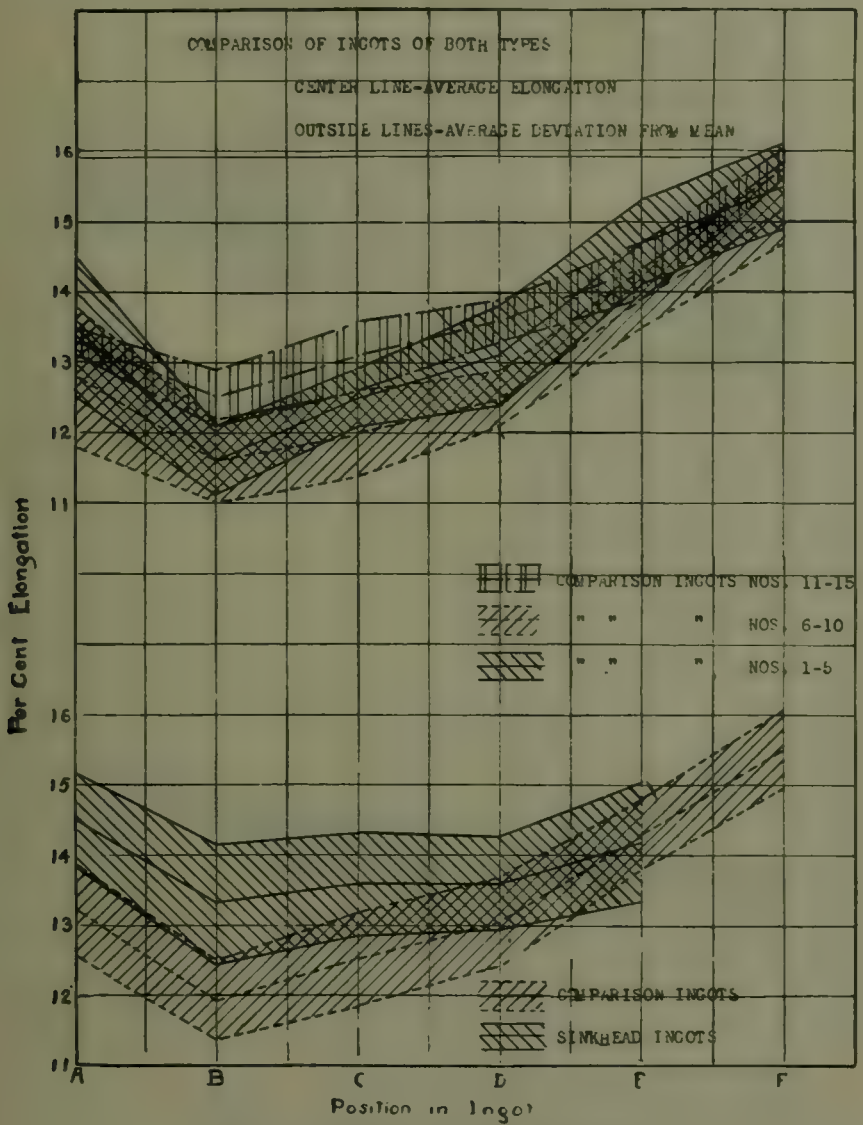


FIG. 15.—*Elongation of ingots of both types*

run closely parallel through the ingot, with M6–M10 having slightly higher ultimate strength. Group M11–M15 is characterized by the abnormally high value (approximately 130 000 pounds per square inch) in the A and B positions.

The yield point shows, in general, characteristics similar to the ultimate strength; for the former, however, there is a somewhat greater uniformity throughout the ingot for all types, and also for

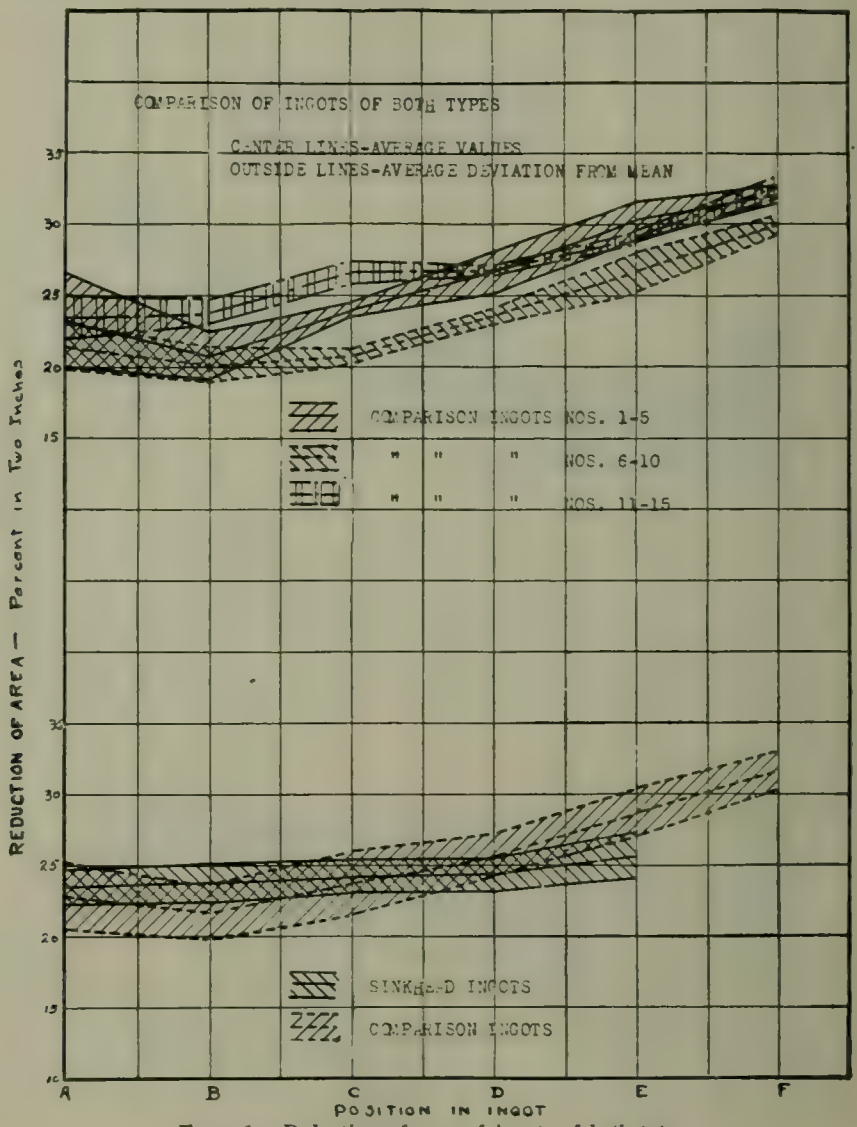


FIG. 16.—*Reduction of area of ingots of both types*

the ingots of a given group. The sink-head ingots have a slightly higher yield point (62 370 pounds per square inch) than the comparison ingots (61 370 pounds per square inch). For the three groups of the latter there is a falling off throughout the length of the ingot from approximately 64 000 pounds to 59 000 pounds per

square inch (as for the ultimate strength), while for the sink-head ingots the yield point remains practically constant throughout.

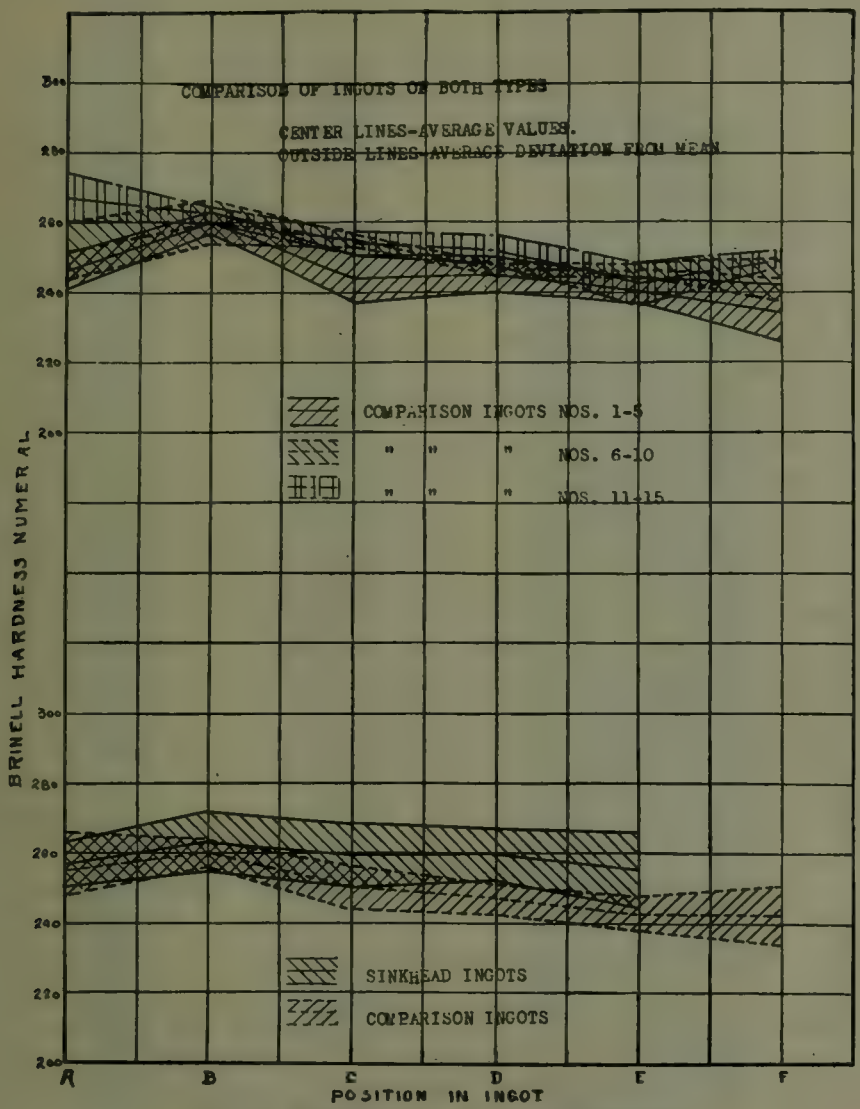


FIG. 17.—*Brinell hardness of ingots of both types*

(*c*) ELONGATION AND REDUCTION OF AREA.—The distribution of ductility, as measured by elongation in tension-test pieces from rails through the length of the sink-head ingots, is somewhat more uniform than for the comparison ingots. The elongation curves are practically the reciprocal of the tensile-strength curves.

The average elongation of the rails from the sink-head ingots is 13.8 per cent, of the comparison ingots 13.6 per cent; the reduction of area for the former type of ingot is 24.38 per cent, for the latter type it is 25.78 per cent, with a range of 21.73 per cent at B to 31.68 per cent at the F position. There are also considerable differences among the three groups of comparison ingots.

(*d*) HARDNESS.—The scleroscope hardness numbers do not appear to have any significance in defining any differences in qualities or properties in the steel of the several ingots. The material from the sink-head ingots have an average hardness of 31, while the steel from ingots cast in the usual manner has an average hardness of 32.

The Brinell numerals are, as would be expected, fairly closely proportional to the ultimate strengths, and they appear to follow even more exactly the yield points. Whether or no there is any definite relation between carbon segregation and Brinell numerals appears difficult to say. The holes for obtaining drillings for the former are ½ inch in diameter and not always exactly coincident in location with the Brinell impression. In a general way one would expect the two to bear some relation to each other. The rails from the sink-head ingots had an average Brinell hardness of 260 (deviation ±8), while the rails made from ingots cast with the small end uppermost without sink heads had a Brinell hardness of 250 (deviation ±6).

(*e*) DROP TESTS.—In the drop tests blows were given on head of rail to destruction, the deflection was measured for each blow, the elongation for each inch over 6 inches and the total elongation noted.

The drop-test piece was taken from near the top of the ingot, and in each case was the first 5-foot length of rail immediately after reaching physically sound steel; that is, free from pipe and in the case of the rails from sink-head ingots also below the bottom of the sink head. Of the 14 comparison rails none broke under 4 or 5 blows (6 rails were nicked and broken after the fourth or fifth blow), while for the 35 Hadfield rails the range was 2 to 7 blows, there being 9 requiring 4 blows, and 4 requiring 3 blows; only 22, or 63 per cent, of the rails from sink-head ingots withstood 4 or more blows. The ductility as measured by the deflection and elongation is also somewhat greater and more uniform for the comparison rails.

The greater uniformity of the sink-head over the comparison ingots, as shown in nearly all other mechanical tests,

FIG. 5.—*Photograph of sulphur print of top bloom from comparison ingot No. 10*

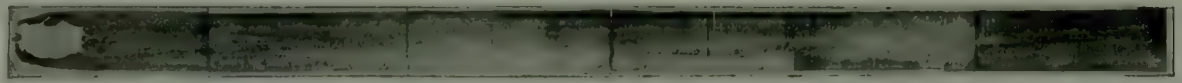


FIG. 6.—*Photograph of sulphur print of top bloom from sink-head ingot No. 5*

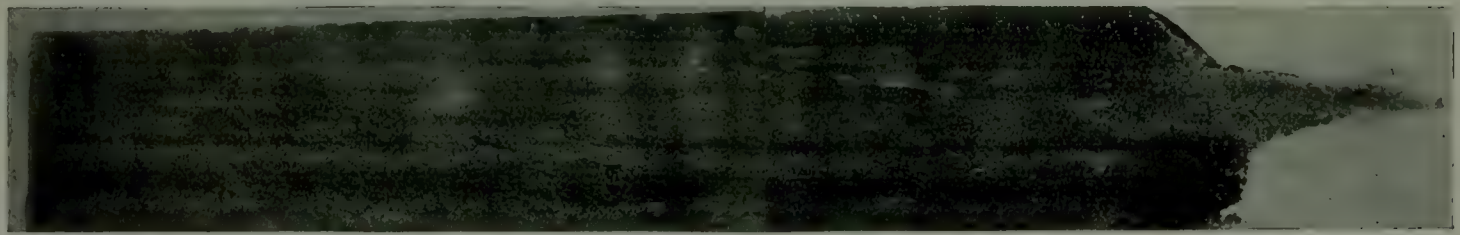
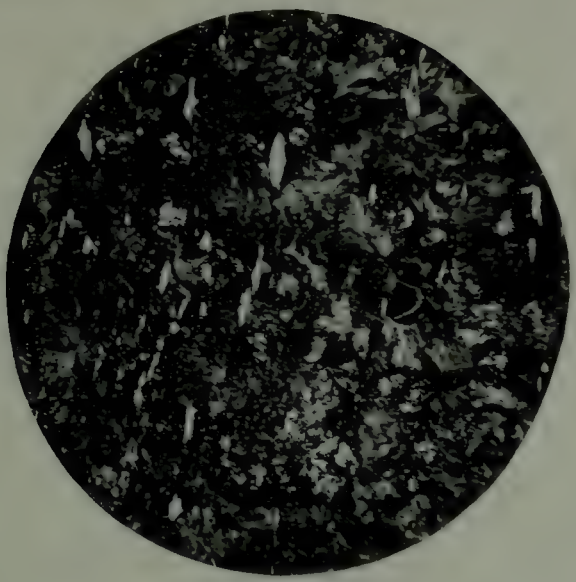


FIG. 7.—*Photograph of sulphur print of half section of sink-head ingot No. 32*

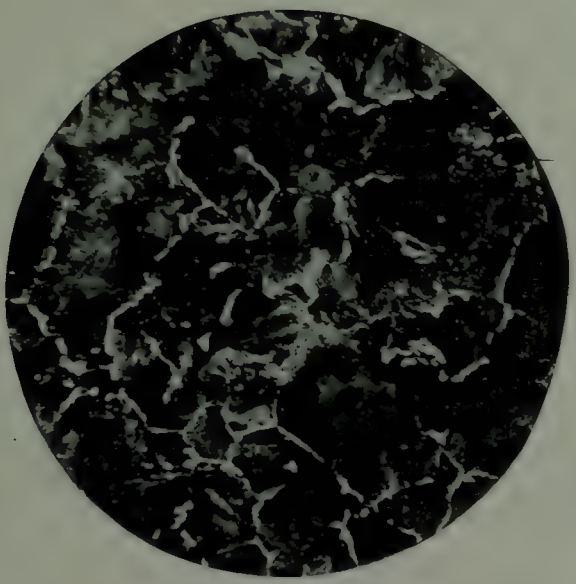
48

FIG. 8.—*Rail M1–A from comparison ingot*

Sulphur print, showing extreme segregation in rail from top of ingot. × 1



a



b

FIG. 8.—*Rail M1–A from comparison ingot (continued)*

Typical microstructure of the metal of rail M1–A at positions indicated in sulphur print. × 50, reduced from × 100

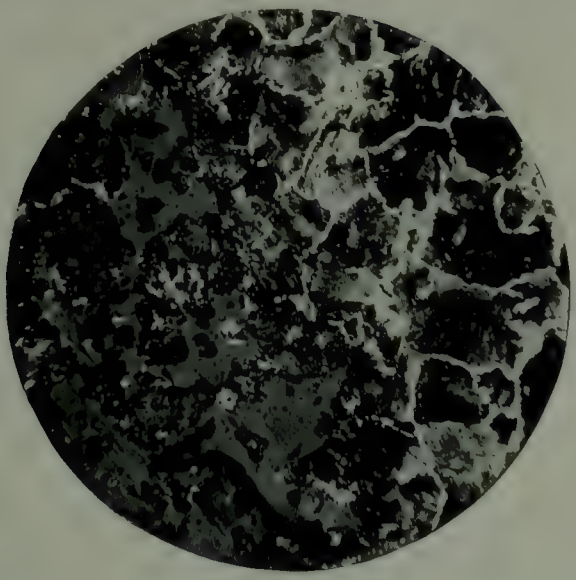
(*a*) Moderately finely grained hypereutectoid steel, pearlite with a trace of free cementite and many small slag inclusions

(*b*) Moderately finely grained hypoeutectoid steel, pearlite with a thick irregular ferrite network

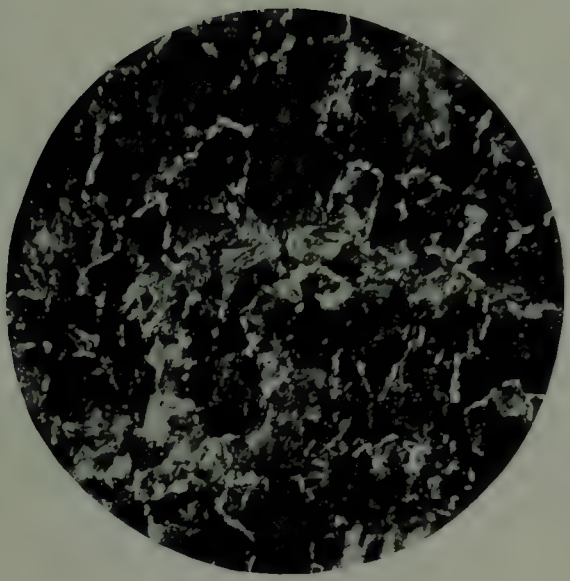
FIG. 9.—*Rail M5–F from comparison ingot*

Sulphur print, showing pipe in rail from bottom portion of ingot (F position). × 1

48 3



a



b

FIG. 9.—*Rail M5–F from comparison ingot (continued)*

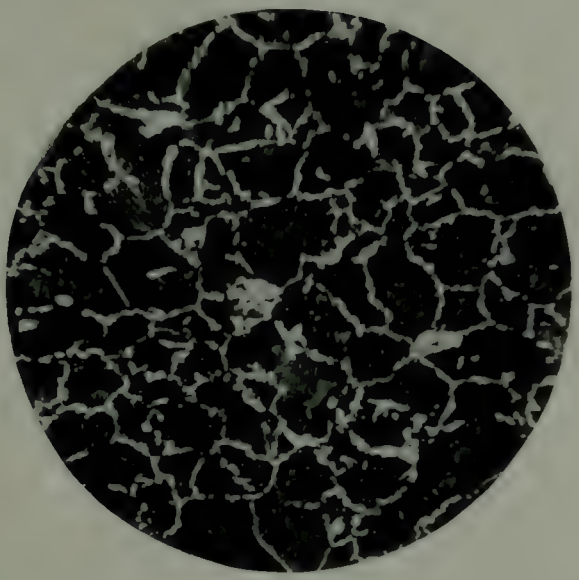
Typical microstructure of the metal of rail M5–F at positions indicated in the sulphur print. × 50, reduced from × 100

(*a*) Structure at the edge of a pronounced pipe, consisting of eutectoid steel contaminated with slag and bordered by a layer of hypoeutectoid metal

(*b*) Moderately finely grained metal, consisting of pearlite with a trace of ferrite, and containing some slag intrusions

FIG. 10.—*Rail M4–F from comparison ingot*

Sulphur print, showing soft streak in web at F position (bottom of ingot). × 1



a

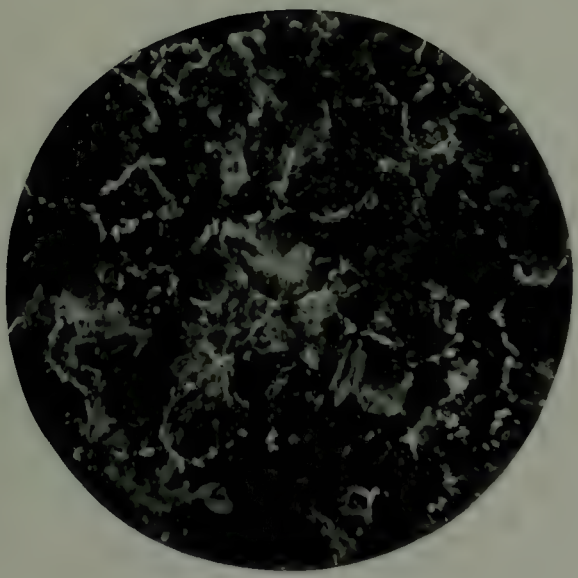


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FIG. 10.—*Rail M4-F from comparison ingot (continued)*

Typical microstructure of the metal of rail M4-F at the positions indicated in the sulphur print. × 50, reduced from × 100

(*a*) Streak of soft metal in the web, consisting of pearlite crystals surrounded by well developed ferrite envelopes

(*b*) Moderately finely grained metal consisting of pearlite and a trace of ferrite

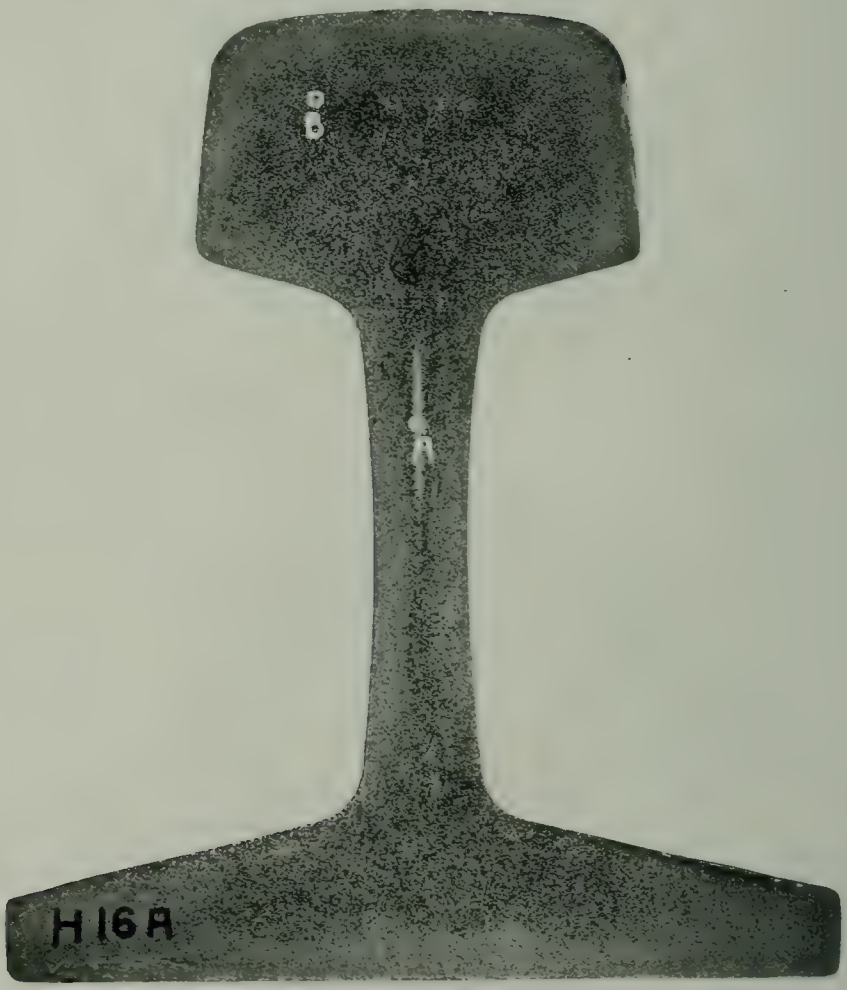
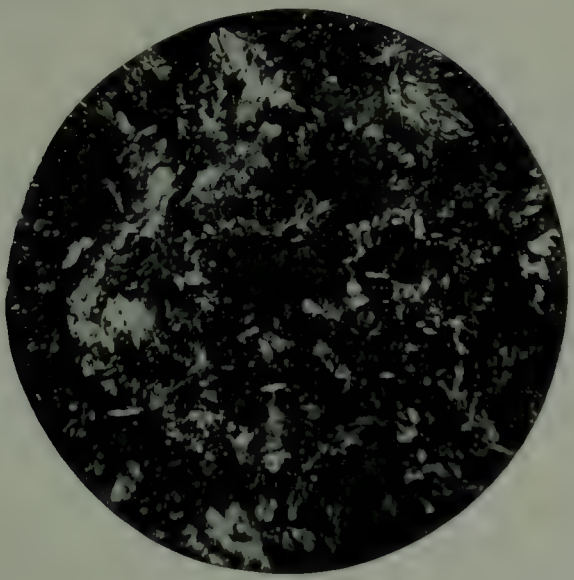
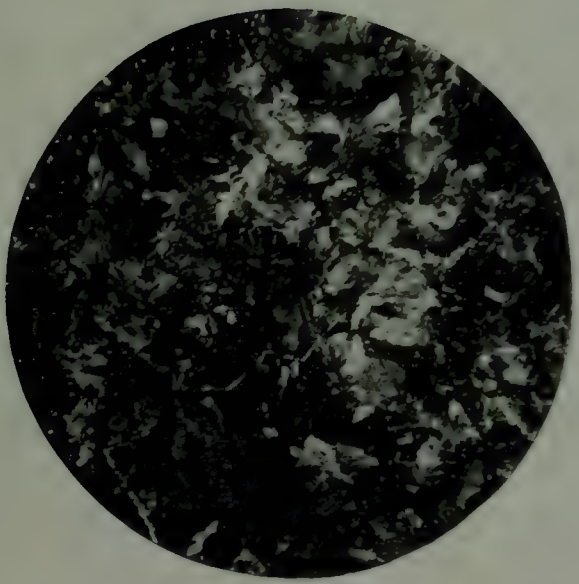


FIG. 11.—*Rail H16–A from sink-head ingot*

Sulphur print, showing slight segregation in upper part of ingot (A position). × 1



a



b

FIG. 11.—*Rail H16–A from sink-head ingot (continued)*

Typical microstructure of the metal of rail H16-A at positions indicated in the sulphur print. × 50, reduced from × 100
(*a*) Eutectoid metal comprising a slightly segregated streak in the web of the rail
(*b*) Average finely grained structure, pearlite with a trace of ferrite

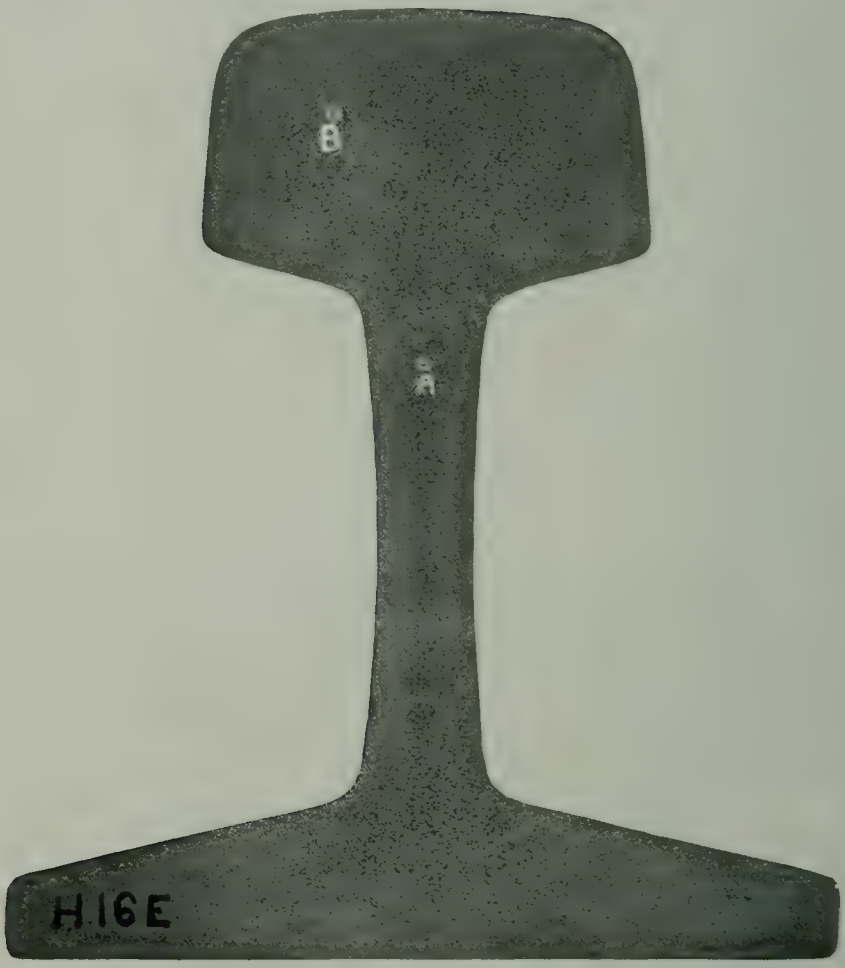
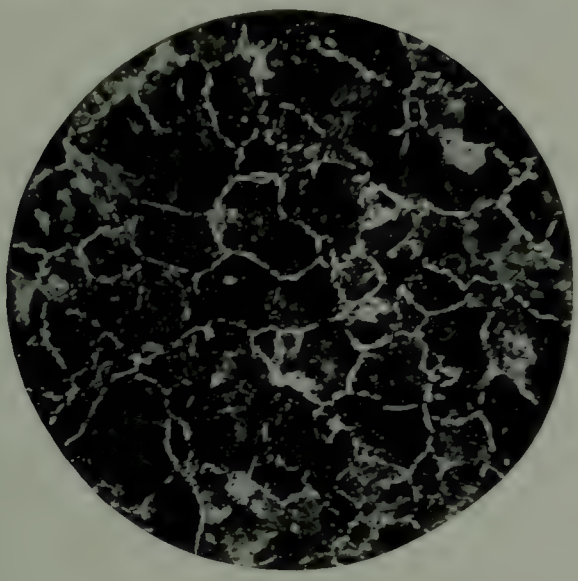


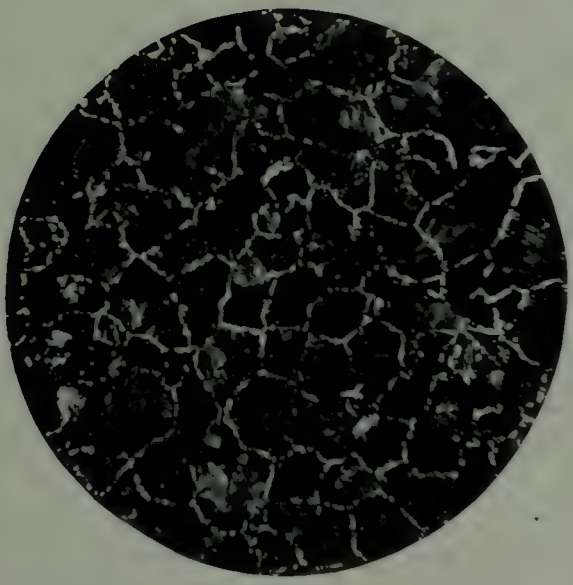
FIG. 12.—*Rail H16–E from sink-head ingot*

Sulphur print, showing sound steel in rail from bottom of ingot (E position). × 1. The rail is homogeneous

48 6



a



b

FIG. 12.—*Rail H16–E from sink-head ingot (continued)*

Typical microstructure of the metal of rail H16–E at the positions indicated in the sulphur print. × 50, reduced from × 100
(*a*) Moderately finely grained metal showing a thick ferrite network
(*b*) Finely grained metal, pearlite with a ferrite network

in less segregation, greater homogeneity as shown by sulphur prints and etching, less amount and variability in discard to sound steel, suggests that the question of the significance and interpretation of the drop test may well be raised with propriety. The greater ductility and more uniform behavior under the drop test of the steel from the comparison ingots would appear to be due more to the chemical composition, and especially to the nickel and chromium content of this Mayari steel, than to any factor traceable to manufacture, which in the case of the comparison ingots is seen to divide them sharply into three groups of very distinct physical characteristics—each a heat—which fact, the drop test is unable to reveal.

8. METALLOGRAPHIC TESTS

The metallographic tests throw additional light on the question of segregation and soundness, and especially with reference to the presence of slag inclusions, streaks, and seams. They also furnish evidence as to the uniformity of the operations in the rolling mill, although there appears also to be a distribution of structure for each ingot characteristic of its manufacturing processes preceding the rolling.

The smoothness and evenness of tone of the sulphur prints of all rail sections from the Hadfield sink-head ingots, which are characteristic of the lot (Figs. 11–12), even from sections in the drop-test portion near the top of the ingot, is in striking contrast to the markedly irregular sulphur prints from comparison rails (Fig. 8), except for a few below the center of some of these ingots. Most of the rails from the Maryland ingots show very pronounced irregularities in terms of the sulphur prints, some of them from the bottom of ingot showing well-defined pipes (Figs. 9 and 10).

As to the microstructure of the steel, here again the sink-head ingots show much the greater uniformity. This is especially true for the upper portions of the ingot, where differences would be expected.

The parallelism of segregation and type of structure is, in general, very close. Thus in the extreme top portion of the ingot there appears a fine-grained, hypereutectoid structure in the segregated area. The main differences in structure across the section, except for these extreme cases accompanying marked segregation, are usually limited to differences in grain size, pearlite with a ferrite network, the smaller-sized grain accom-

panying the higher carbon. This is the predominant characteristic of the microstructure of the rails from the sink-head ingots.

The rails from the comparison ingots have, below the segregated area, about the same structural characteristics as those from the sink-head type, except that for many of them the ferrite network is less pronounced and in some cases almost absent. The rolling and finishing temperatures of the ingots were not widely different and do not appear to play any considerable rôle in determining differences of structure. The manganese content, usually somewhat higher in the Hadfield ingots, apparently tends to accentuate the sharp, definite boundaries of ferrite surrounding the pearlite grains. A number of typical photomicrographs are shown in Figs. 8–12, inclusive.

9. EXAMINATION OF SPLIT INGOTS AND BLOOMS

(*a*) SINK-HEAD INGOT H37.—Sink-head ingot H37 was cut in halves at Sheffield and Sir Robert Hadfield reports:

> The whole of this ingot could be worked into rails after cutting off 10 per cent discard. The ingot is perfectly sound and represents the average quality of the whole of the 36 ingots sent to be rolled into rails.
>
> The capacity of the sink-head cavity of this ingot was 8125 cubic centimeters, and the percentage of settling, by weight in pounds—that is, of the total weight of the ingot—was 2.58 per cent. The weight of the material which passed from the head portion into the ingot itself was about 130 pounds. This ingot was selected because it showed the lowest cavity percentage; namely, 2.58 per cent.

(*b*) SINK-HEAD INGOT H32.—Sink-head ingot H32 was cut in halves at the Bureau of Standards. A photograph of a sulphur print of a longitudinal half section of the ingot is shown in Fig. 7. In Figs. 18–21, inclusive, are shown the location of drill holes for chemical analyses of this ingot, as well as the results of the analyses. Only carbon, phosphorus, silicon, and sulphur surveys were made, as the rail analyses indicated that there was no appreciable segregation of manganese in similar ingots. The results obtained indicate that with about 9 per cent discard, steel free from piping and appreciable segregation is obtained. Carbon showed a high degree of segregation (0.68 to 1.13 per cent), but as will be noted, this is confined to the extreme upper portion of the ingot. The high carbon content at this point is probably due to the fact that the steel in the top part of the ingot absorbed carbon from the charcoal used, together with air blast, to keep the ingot hot on its upper surface while cooling. Phosphorus, silicon, sulphur, and manganese are remarkably uniform in this sink-head type of ingot.

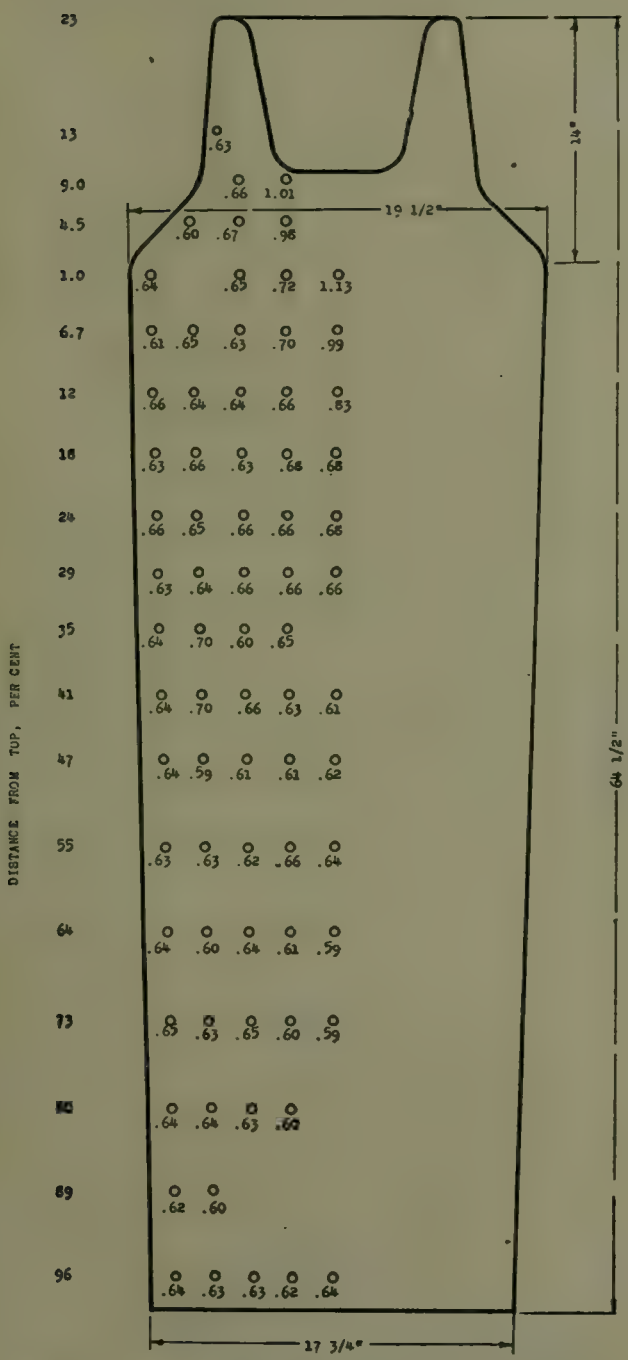


FIG. 18.—*Section of Hadfield split ingot No. 32, showing percentage of carbon*

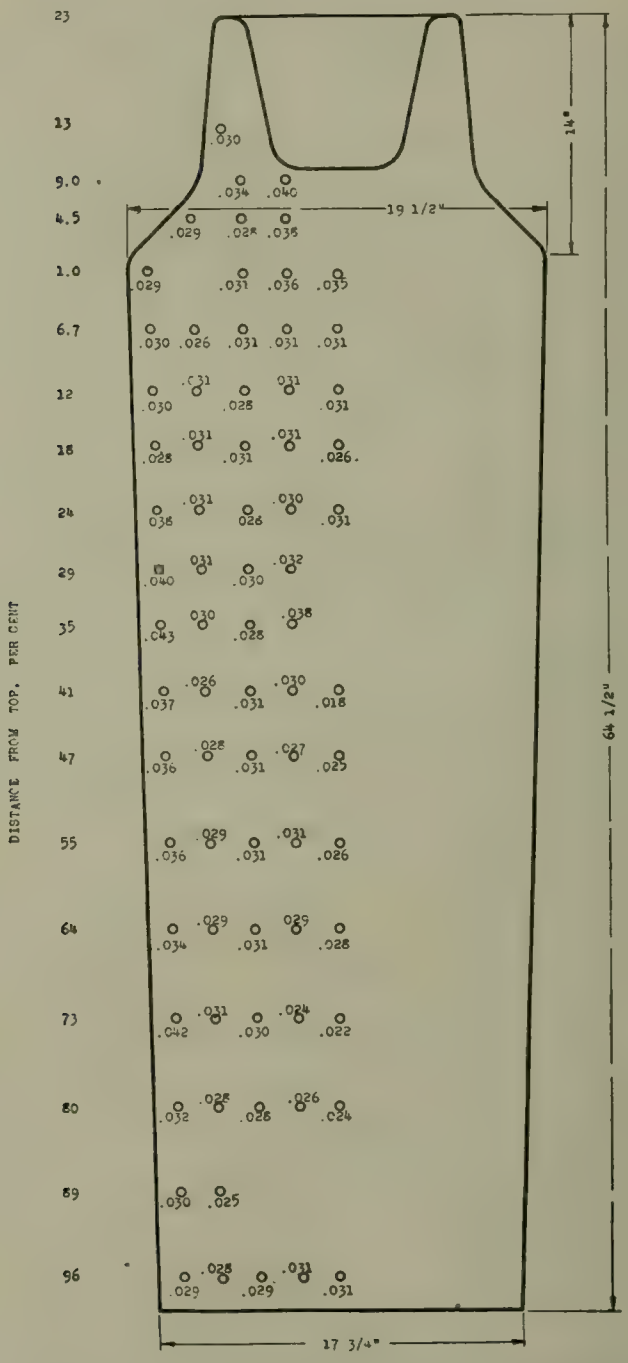


FIG. 19.—*Section of Hadfield split ingot No. 32, showing percentage of phosphorus*

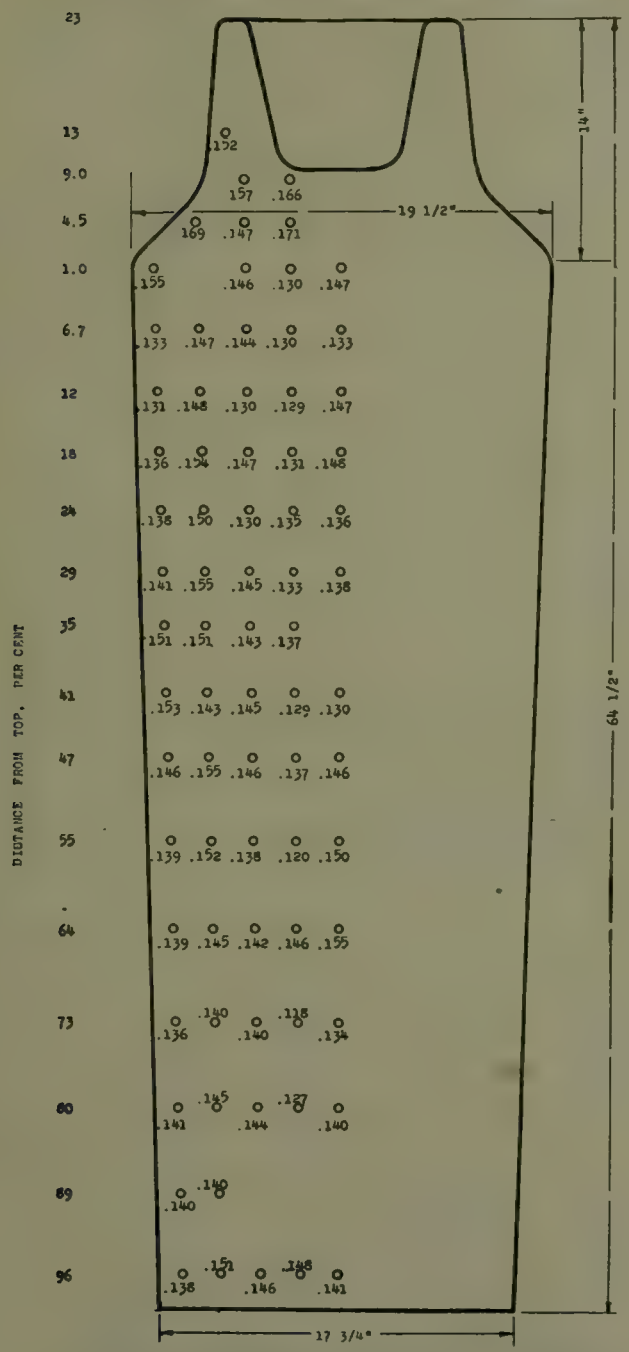


FIG. 20.—*Section of Hadfield split ingot No. 32, showing percentage of silicon*

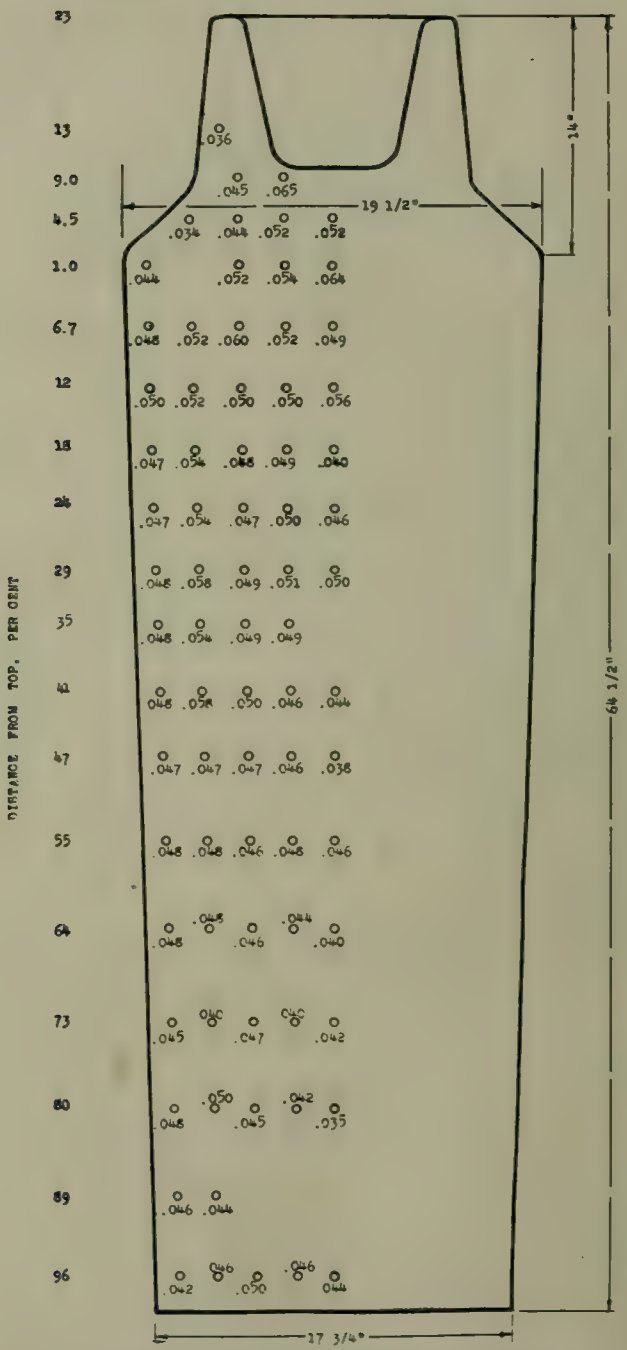


FIG. 21.—*Section of Hadfield split ingot No. 32, showing percentage of sulphur.*

(*c*) SINK-HEAD INGOT H5 AND COMPARISON M10 TOP BLOOMS.—The top blooms from ingots H5 and M10 were split longitudinally at the Bureau of Standards and one half of each used for sulphur prints (Figs. 5 and 6) and the other half of each for a chemical survey (Figs. 22 and 23) of carbon, phosphorus, silicon, and sulphur segregation.

As in the case of the sulphur prints from the rails, the sulphur print from the bloom of the sink-head ingot is of even tone, while the comparison bloom shows decided irregularities (Figs. 5 and 6). In addition, the latter bloom has an inclosed pipe some 12 per cent below the top.

The chemical survey shows the Hadfield bloom to be very uniform. The maximum carbon is 0.90 per cent, and this is only 4 per cent from the top of the ingot. The comparison bloom, according to chemical analyses, is also quite uniform except for the sulphur content, which is very irregular. This element attains a maximum of 0.166 per cent at a distance of 12 per cent from the top, while it is as high as 0.12 per cent at a distance of 21 per cent.

10. SPECIAL NICKEL CHROMIUM SINK-HEAD INGOT

A special sink-head ingot, of the same dimensions and type as the other sink-head ingots but containing chromium and nickel was rolled into rails so as to permit more accurate comparison with the ingots cast in the ordinary manner. The latter naturally contain small amounts of nickel and chromium from the Mayari ore from which the product was made.

The mechanical and chemical properties of the five rails from this special ingot (H38) are given in Table 23. It will be observed that the yield point (70 791 pounds per square inch), the ultimate tensile strength (143 025 pounds per square inch), and the Brinell hardness (292) are greater, while the elongation (11.3 per cent) and reduction of area (19.06 per cent) are less than those of the average sink-head ingot. Under the drop test the rails from this ingot behave similarly to the comparison ingots.

From the standpoint of composition the steel from this ingot differs from that of the average sink-head ingot in that it is of higher carbon (0.73 per cent) and lower phosphorus (0.016 per cent) and sulphur (0.018 per cent); also in that it contains small amounts of nickel (0.19 per cent) and chromium (0.11 per cent).

It was necessary to discard 17.6 per cent of this ingot because of pipe, as against 9.1 per cent for the average sink-head ingot.

TABLE 23.—Mechanical and Chemical Properties of the Special Sink-Head (Nickel and Chromium) Ingot

Rail	Tension tests				Hardness	
	Yield point	Ultimate strength	Elongation, per cent in 2 inches	Reduction of area	Brinell	Scleroscope
	Lbs./in.²	Lbs./in.²		Per cent		
H38-A	70 030	142 725	12	18.79	274	33
H38-B	70 700	143 400	11	18.59	298	32
H38-C	70 950	143 450	12	18.54	318	29
H38-D	71 445	143 300	11.5	18.38	283	33
H38-E	70 830	142 250	10	21.00	287	31
Average	70 791	143 025	11.3	19.06	292	32

Rail	Results of chemical analysis at "O"; per cent								
	Carbon			Mn	P	Si	S	Ni	Cr
	O	M	Seg.						
H38-A	0.741	0.741	[illegible]	0.91	0.024	0.179	0.018	0.19	0.10
H38-B	.731	.729	−0.27	.95	.027	.177	.019	.19	.10
H38-C	.728	.735	.96	.93	.026	.179	.018	.19	.12
H38-D	.734	.726	−1.09	.94	.027	.185	.018	.18	.10
H38-E	.731	.730	− .14	.96	.025	.180	.019	.19	.11
Average	.733	.732	− .11	.94	.026	.180	.018	.19	.11

IV. SERVICE OF RAILS FROM THIS INVESTIGATION

The Pennsylvania Railroad, through A. W. Gibbs, reports as follows on the service of the rails from the earlier investigation [6] and from the present one:

In January, 1915, the Maryland Steel Co. shipped free for test 5 No. 1 33-foot P. S. 100-pound open-hearth steel rails rolled from ingots furnished by Sir Robert Hadfield to the Bureau of Standards. These rails were laid in the Eastward freight track on Horseshoe Curve, January 20, 1915, and removed May 15, 1915. The average abrasion of the Hadfield rails was 1.16 square inches and that of the ordinary rail 0.78 square inch. None of these rails failed.

In September, 1915, the Maryland Steel Co. rolled 46 No. 1 and 61 No. 2 P. S. 100-pound rails (0.33 track mile) from imported Hadfield sink-head ingots and at the same time rolled 53 No. 1 and 4 No. 2 P. S. 100-pound rails (0.18 track mile) from its own ingots for comparison. The 46 No. 1 Hadfield and 53 No. 1 Maryland special rails were laid November 26, 1915, in the Eastward passenger and freight track on the 5° 30′ curves between Spruce Creek and Union Furnace, Middle Division, and removed October 23, 1916. The 61 No. 2 Hadfield and 4 No. 2 Maryland special rails were laid on a 2° curve in the Eastward Low Grade track, Philadelphia Division, west of M. P. 69, and are still in track. The average abrasion of the No. 1 Hadfield rails was 0.42 square inch; that of the Maryland special rails 0.37. The rail on Low Grade was still in track October 31, 1919; abrasion was then 16.1 per cent for Hadfield and 9.7 per cent for Maryland special.

There have been no failures of Hadfield rails or of the Maryland specials.

There is evidently no economy in the Hadfield process as regards wear, and it would require a much more extended trial to determine the relative freedom from failure.

[6] See footnote 1.

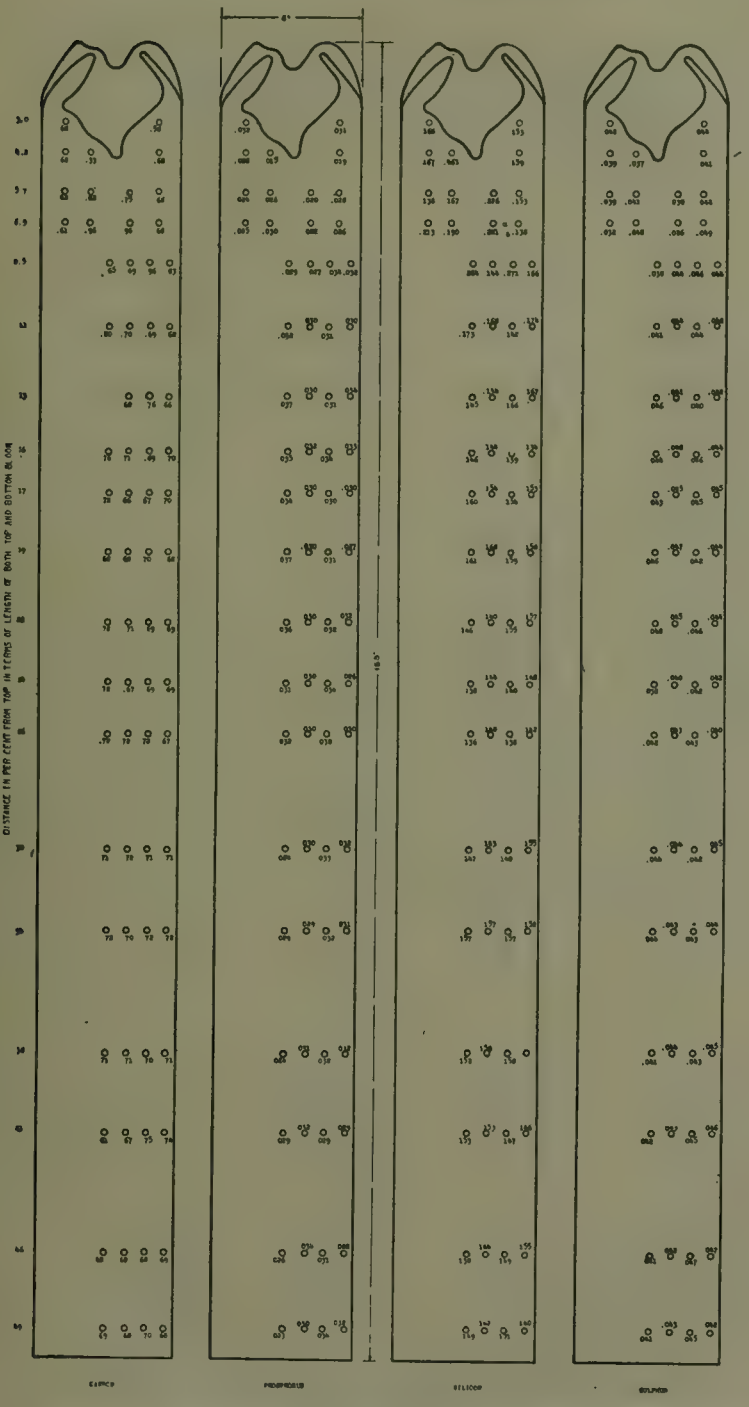


FIG. 22.—*Sections of top bloom from Hadfield ingot H5, showing chemical analysis of various positions*

FIG. 23.—*Sections of top bloom from Maryland ingot M10, showing chemical analysis of various positions*

The Maryland rails may owe their less abrasion to their content of nickel and chromium. It would appear to be unfair to draw any general conclusions as to performance in service from so few rails as this investigation furnished, although it would not be expected that the Hadfield type of sink-head ingots would furnish any rails showing structural defects.

V. SUMMARY AND CONCLUSIONS

The object of this investigation was to determine the relation of ingot practice to the properties of rails from such ingots, and in particular the amount of total discard necessary to obtain rails free from piping and segregation above 12 per cent which had been rolled from steel made in accordance with varying melting, casting, and ingot practices.

To that end, 35 ingots made by the converter process at Hadfield's, Sheffield, England, and cast by the sink-head process with large end uppermost were shipped to Sparrows Point, Md., and rolled into rails in comparison with 15 rail ingots made in the ordinary manner with the small end uppermost. Each sink-head ingot, of 5300 pounds weight, and deoxidized with aluminum in the mold, represented a separate heat of converter steel, and all the heats and ingots were made in the same manner. The composition and properties of these ingots were of remarkable uniformity. The comparison ingots, of 7300 pounds each, were from 3 separate open-hearth heats, an intentional variation being made in the open-hearth and casting practice for each. Five ingots were selected from each of these 3 heats. Thus in reality, comparison was made of 4 different kinds of steel, of very nearly the same composition and physical properties and of two types of ingot form.

The comparison was made by rolling most of the ingots into rails and taking test specimens at each rail cut, as well as from a considerable portion of the upper part, in 5-foot steps, of the rail bar from each ingot. In this way there was obtained a detailed physical, chemical, and metallographic survey of each ingot, and it was possible to exactly delimit the regions of sound and homogeneous from those of unsound and segregated steel. Two complete sink-head ingots were cut longitudinally and examined, as well as representative blooms from both sink-head and ordinary ingots.

The results obtained indicate a decided superiority of the sink-head ingots over the comparison ingots as made of three grades of steel (Tables 17 and 18), although the sink-head ingots suffered from the disadvantage of having gone cold before rolling. The

Hadfield type of ingot required a total discard of only 18.4 per cent on the average (13 per cent top discard to eliminate piping and segregation above 12 per cent) while the average ingot of the ordinary type for rails required a total discard of 43.9 per cent (26 per cent top discard), with great variations dependent upon the furnace and ingot practices.

The comparison ingots from heat M1–M5 of nondeoxidized rising steel, chilled on top of ingot by cast-iron caps, required excessive discard to eliminate positive segregation at the top and negative segregation at the bottom of the ingot, the latter often accompanied by dangerous inclosed pipes.

The second heat (M6–M10) made of rising steel deoxidized with aluminum in the molds; the ingot tops of which were cooled with water, required the least total discard of the three heats. It was more subject to piping and less to segregation than the first heat of ingots made in the usual manner.

The third heat (M11–M15) made of quiet or "killed" steel, was not chilled on top with water or caps, and was deoxidized with aluminum in the molds. The ingots of this heat required an intermediate amount of total discard when compared to the first and second heats; this heat was the only one for which a greater top discard was required to eliminate piping than to eliminate segregation above 12 per cent. One of the ingots of this third heat contained a small pipe at the bottom, and all the rails from the middle and bottom of the ingots showed high negative segregation.

The variation of physical properties throughout the length of each ingot is characteristic not only of the type of the ingot, as sink-head or ordinary, but also of the state of the steel when cast and of the ingot practice.

It has been established in the foregoing that, after removal of the top discard of 13 per cent, the Hadfield type of sink-head ingot is free from piping and undue segregation. The ordinary type of ingot, cast small end up without sink head, as is usual for rail ingots, requires an average top discard of 26 per cent, and the remainder of the ingot is liable to contain inclosed piping and excessive segregation. Defective rails, from the middle and bottom portion of the ingot, are not detected with certainty by means of the test for existing rail specifications, and as a result of this uncertainty rails containing pipes or excessive segregation may get into service with disastrous results.

The surface condition of the rails from the sink-head ingots was not as good as that of the ordinary ingots, but this is not considered an essential characteristic of rails from such ingots.

The markedly differing characteristics of the three heats of comparison ingots lead one to raise the question whether or not it might be advisable to specify, at least in some degree, the methods of steel manufacture or of ingot practice for rails and similar products on which the safety of the traveling public depends

While it is not claimed that the use of the sink-head process for the manufacture of ingots will solve all rail problems, it is maintained that its adoption would be a step in the right direction in view of the present heavy casualties and property losses on American railroads. The necessary changes in mill operations, it is believed, could be made without too great difficulties.

The service results, showing no failures to date, in the few rails of this investigation can not be considered conclusive one way or another.

It would be of the greatest practical importance in tracing statistically the origin of rail failures, if, in the reports to the rail committee of the American Railroad Engineering Association, they be grouped with reference to their position in the ingot, and in addition the steel manufacturing process be reported, at least briefly by such notations as rising steel capped with plates; quiet steel deoxidized with x ounces of aluminum per ton, etc. If then it be found, for a given kind of steel, there is a greater or less tendency for certain types of failure to predominate or to exist in certain definite regions in the ingot, corrections could then be made intelligently in the manufacturing process. The results of the present investigation indicates very strongly that one should expect the four types of steel and two types of ingot examined, in view of their sharply differing characteristics, to behave differently in service.

WASHINGTON, June 7, 1920.

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BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 179

ELECTRIC-ARC WELDING OF STEEL:
I. PROPERTIES OF THE ARC-FUSED METAL

BY

HENRY S. RAWDON, Physicist
EDWARD C. GROESBECK, Physical Chemist
LOUIS JORDAN, Associate Chemist
Bureau of Standards

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-Note- 2a

Doubt has arisen from the results of some special etching tests, described by C. F. Comstock in his discussion of "Nitrogen in Steel, and the Erosion of Guns", H. E. Wheeler, Trans. Amer. Inst. of Mining and Met. Engrs., 1920, and tried out by the authors several months following the preparation of this paper, as to the correctness of the identification of the microconstituent designated as "pearlite islands" (See Figs. 21e and 24e), The strong similarity in appearance of these "pearlite islands" to the pearlite in ordinary low-carbon steels, as developed by the usual etching reagents, picric acid, nitric acid, etc., and apparent compatability of the carbon content of the arc-fused iron with the extent of the "pearlite islands" noted, naturally led to the assumption that this constituent was pearlite, particularly as there was found in many of the "pearlite islands" in the slowly-cooled samples a border that extended part way around the edge very similar to cementite atolls in partially divorced pearlite. It had been noted, however, that these "pearlite islands" possessed in many cases a rather more angular-shaped appearance than is generally found.

The results of the above-mentioned tests failed to prove these "pearlite islands" to be really pearlite as usually formed in carbon steels. It might appear from the similarity in behavior during etching to that of the needles or plates (assumed to be iron nitride), that this constituent owes its existence to the prese nce of nitrogen, though there is no clear proof for this. Whether the small amount of carbon, as indicated by chemical analysis to be present in the metal, had a contributory influence upon the formation of this constituent or not is not clear. Wilson (Chem. & Met. Chem. 23, p. 1161, 1920) definitely states that this pearlite-like constituent is an iron-iron nitride eutectoid though no substantiating evidence is given.

December 29, 1920.

ELECTRIC-ARC WELDING OF STEEL: I. PROPERTIES OF THE ARC-FUSED METAL

By Henry S. Rawdon, Edward C. Groesbeck, and Louis Jordan

CONTENTS

	Page
I. Introduction	3
II. Aim of the investigation	4
III. Preliminary examination of electric-arc welds	5
IV. Method and materials	7
V. Changes in composition of electrodes as a result of fusion	11
VI. Mechanical properties of the arc-fused metal	14
VII. Structural features: Metallographic examination	23
1. Macrostructure	23
2. Microstructure	26
(*a*) General features	26
(*b*) Microscopic evidence of unsoundness	29
(*c*) Characteristic "needles" or "plates"	31
(*d*) Relation of microstructure to the path of rupture	36
3. Effect of heat treatment upon structure	40
4. Thermal analysis of arc-fused steel	48
VIII. Discussion of results	54
IX. Summary	57
Appendix—Bibliography	60

I. INTRODUCTION

The daily advances in the variety and number of applications of electric welding bespeak the continually increasing industrial importance of this method of joining and repairing metals. Of the two forms of electric welding in common industrial use, the arc-fusion method is of far more general application than is electrical-resistance welding; arc welding, as is evident from its nature, can be applied in a multitude of ways to as many different welding projects, while the second method, which depends upon the contact resistance of the two parts which are to be joined together, finds its greatest applications in "repeat" welding.

Resistance welding is closely related to the familiar weld of the smith's forge in that the two surfaces to be joined are heated to the welding temperature, the electric current being the source of the necessary heat, and then pressed into intimate contact while

hot. The principle of arc welding, however, is very different; the two parts are joined together by fusion, the fused metal being supplied from an outside source rather than from the parts which are to be welded. Often the addition of a very considerable amount of metal is necessary, which metal is supplied by the fusion accomplished by means of the heat of the electric arc. A layer of considerable thickness often exists between the two parts joined together.

II. AIM OF THE INVESTIGATION

The application of arc welding in the shipbuilding industry is one of the most important and extensive applications which has as yet been proposed for this method. During the year 1918, at the request of and with the cooperation of the Welding Research Subcommittee of the Emergency Fleet Corporation, an extensive program was outlined by the Bureau of Standards for the study of this type of welding. The principal object of the research was to determine in an empirical way by actual welding tests carried out by skilled operators the relative values and merits of the different types of electrodes or welding pencils which are available commercially. Due to changed conditions, however, at the close of the year 1918 the original program was modified and shortened very considerably. The results of the investigation here given relate principally to the nature and characteristic properties of the weld, and in particular those of the 'fused-in" metal.

In drawing up the modified program it was decided to make the study of the characteristic properties of the "fused-in" metal the primary object of the investigation, the study of the merits of the different types of electrodes being a secondary one. Since the metal of any weld produced by the electric-arc fusion method is essentially a casting because there is no refinement possible as in some of the other methods, it is apparent that the efficiency of the weld is dependent upon the properties of this arc-fused metal. Hence a knowledge of its properties is of fundamental importance in the study of electric-arc welds.

Though the results obtained throughout the entire series of examinations are not so conclusive as might be desired, it is believed that they contribute very materially to our knowledge of the properties of the "fused-in" metal, which conditions the serviceability of the electric-arc weld.

III. PRELIMINARY EXAMINATIONS OF ELECTRIC-ARC WELDS [1]

Numerous articles have appeared in the technical literature bearing on the subject of electric-arc welding. Most of these, however, are devoted to the technique and comparative merits of the method, manipulations, equipment, etc., rather than to the study of the characteristics of the metal of the weld itself. The information on this phase of the subject is rather meager.[2]

A considerable number of examinations was made of welds prepared by means of the electric-arc process and representative of different conditions of welding. Most of these were of a general miscellaneous nature, and the results do not warrant including a description of the different specimens here. One series of particular interest, however, may well be referred to in detail. As part of this study the Welding Research Subcommittee submitted to the Bureau of Standards for examination a number of welds of ship plate representative of English practice, some of the welds being considered as very superior examples of welding, others being of a decidedly inferior grade. In Tables 1 and 2 are given the results obtained by the mechanical tests made upon these specimens. The welding was done by skilled operators by means of special brands of electrodes (welding pencils), the trade names of which, however, have been omitted from the tables. In addition to the mechanical tests the specimens were examined very carefully microscopically. The results are not included, however, as the structural features of the material did not differ from those discussed later in Section VII. The results of the mechanical tests given are of value in that they are indicative of the average mechanical properties which should be expected in electric-arc welds of satisfactory grade and of the shape and size of those examined.

---

[1] These examinations, as well as the preparation of many of the specimens used later in the investigation, were made by B. Hurvitz, formerly assistant chemist of the Bureau of Standards.

[2] A fairly complete bibliography on the mechanical properties and structure of the metal of the weld and also on nitrogen in iron and steel has been given in the appendix. The necessary references throughout the text are made by quoting the bibliography title number.

TABLE 1.—Mechanical Properties of 12 Good Welds [a]

No.	Average voltage, direct current	Average current	Type of weld [b]	Thickness of plate	Ultimate tensile strength	Elongation	Gage length	Fracture
		Amperes		Inch	Lbs./in.^{2}	Per cent	Inches	
1	60	120	Vertical..	3/4	51 450	7	8	In weld, fine crystalline, some holes
2	60	120	...do.....	3/4	53 200	11	8	Do.
3	[c] 75	110	Flat.....	5/8	57 430	12	8	Outside weld, few holes.
4	[c] 75	110	...do.....	5/8	54 210	14	8	Do.
5	110	70	...do.....	1/4	60 610	5.5	6	Do.
6	110	125	...do.....	3/4	59 000	6	6	In weld, crystalline, fine to coarse, few holes
7	110	150	...do.....	1	52 570	6	6	In weld, very fine crystalline, few holes
8	110	100	Overhead.	1/2	59 470	4	6	In weld, fine crystalline, many holes
9	110	120	Flat.....	1/2	55 460	6	6	In weld, crystalline, fine to coarse, few holes
10	110	120	Vertical...	1/2	49 030	3.7	6	In weld, coarse crystalline, few holes
					Cold-bend test [d]			
					Load at yield	Load at break	Angle of bend	
					Pounds	Pounds	Degrees	
11	110	70	Flat.....	1/4	850	1 010	105	
12	110	150	...do.....	1	13 530	17 450	20	In weld, fine crystalline, few holes

[a] All the welds were made in steel plate of the thickness shown. Electrodes of the covered type were used. The welds were of 60° V type except the overhead welds, in which a 90° V was used.

[b] Refers to the position of the plates which were being welded together.

[c] Alternating current.

[d] The bend tests were made with the apex of V in tension with a 7-inch span over a pin of 2-inch radius except the ¼-inch plate, for which a pin of 1-inch radius was used.

TABLE 2.—Mechanical Properties of 12 Inferior Welds [a]

No.	Average voltage, direct current	Average current	Type of weld [b]	Thickness of plate	Ultimate tensile strength	Elongation	Gage length	Fracture
		Amperes		Inch	Lbs./in.²	Per cent	Inches	
13	110	120	Flat	5/8	32 460	Nil		In weld, fine crystalline, many holes
14	95	105	Vertical	1	19 890	Nil		In weld, spongy metal, poor junction with metal of plate
15	95	105	Flat	1	31 700			Do.
16	60	120	do	1/2	37 290	2	6	In weld, very fine grained with many holes
17	60	110	Vertical	1/2	38 820	2	6	In weld, coarse crystalline, many holes
18	[c] 75	110	do	7/8	31 360	Nil		In weld, fine to coarse crystalline, many holes
19	[c] 75	110	do	1	27 090	1.4	8	In weld, coarse crystalline, many holes
20	110	120	do	1/2	39 400	3.5	6	In weld, very fine grained with crystalline areas, many holes
21	110	120	Flat	1/2	34 650	Nil		In weld, very fine grained, very many holes
22	110	120	do	3/4	35 120	Nil		In weld, very fine to coarse crystalline, many holes
					Cold-bend test [d]			
					Load at yield	Load at break	Angle of bend	
					Pounds	Pounds	Degrees	
23	95	105	do	1	4 930	4 930	Nil	In weld, very many laps and holes
24	95	105	Vertical	1	4 120	4 610	1	Do.

[a] See note [a], Table 1. [b] See note [b], Table 1. [c] See note [c], Table 1. [d] See note [d], Table 1.

IV. METHOD AND MATERIALS [3]

The specimens required for the study of the mechanical properties of the arc-fused iron were prepared for the most part at this Bureau, direct current being used in the operation. The apparatus used is shown diagrammatically in Fig. 1. By means of the adjustable water rheostat the current could be regulated from 110 to 300 amperes. By the use of automatic recording instruments the voltage and current were measured and records were taken at intervals during the preparation of a specimen.

[3] Acknowledgement is due A. B. Engle, formerly laboratory assistant, for aid in the preparation of many of the specimens.

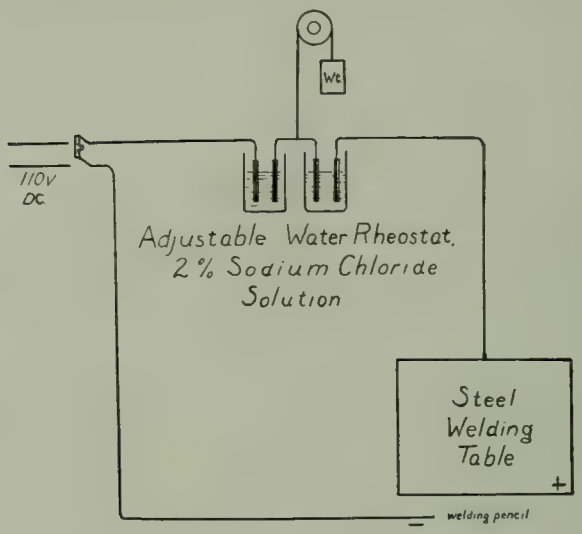


FIG. 1.—*Arrangement of apparatus for welding*

The values of current given below in the tables are those which were desired and were aimed at, the average deviation from the value as recorded by the curves being approximately ±5 amperes.

Since the investigation was concerned primarily with the properties of the arc-fused metal, regular welds were not made. Instead the metal was deposited in a block large enough to permit a tension specimen (0.505 inch diameter, 2-inch gage length) to be machined out of it. Although the opinion is held by some welders that the properties of the metal of an arc weld are affected materially by the adjacent metal by reason of the interpenetration of the two, it was decided that the change of properties of the added metal induced by fusion alone was of fundamental importance and should form the basis of any study of arc welding. The method adopted also permitted the use of larger specimens with much less machining than would have been possible had the metal been deposited in the usual form of a weld.

In the first few specimens prepared (10 in number) the metal was deposited by a series of "beadings" as shown in Fig. 2*a*. The tension specimens cut from the deposited metal were found to be very inferior and entirely unsuitable for the study. This was largely on account of the excessive overheating which occurred, as well as the fact that a relatively "long arc" was

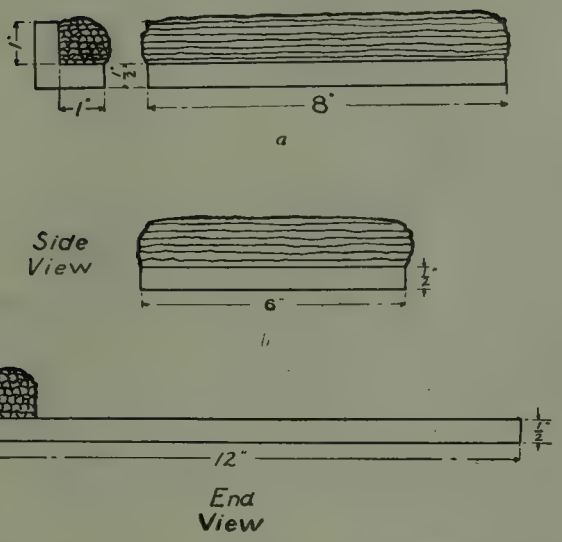


FIG. 2.—*Method of formation of the blocks of arc-fused steel*

a, Early method used, but discarded because of the inferiority of the resulting specimens; *b*, method used in the formation of all specimens, the results of which are given in the following pages

necessary for the fusion in this form. Because of the very evident inferiority of these specimens the results of the mechanical tests made are not given in the tables below (Sec. VI). The method of deposition of the metal was then changed to that shown in Fig. 2*b;* this method also had the advantage in that the amount of necessary machining for shaping the specimens for test was materially reduced. The block of arc-fused metal was built up on the end of a section of one-half inch plate of mild steel (ship plate) as shown. When a block of sufficient size had been formed, it, together with the portion of the steel plate immediately beneath, was sawed off from the remainder of the steel plate. The tension specimen was turned entirely out of the arc-fused metal. No difficulty whatever was experienced in machining the specimens. Fig. 3 shows the general appearance of the block of fused metal as well as the tension specimen turned out of it.

In general, in forming the blocks the fused metal was deposited as a series of "beads" so arranged that they were parallel to the axis of the tension specimen which was cut later from the block. In two cases for purposes of comparison the metal was deposited in "beads" at right angles to the length of the specimen. In all the specimens after the deposition of each layer the surface

was very carefully and vigorously brushed with a stiff wire brush to remove the layer of oxide and slag which formed during the fusion. There was found to be but little need to use the chisel for removing this layer.

FIG. 3.—*Block of arc-fused metal with tension specimen cut from it. Reduction, approximately ½*

Two types of electrodes were used as material to be fused. These differed considerably in composition (Table 3, Sec. V) and were chosen as representative of a "pure" iron and a low-carbon steel. The two types will be referred to as "A" and "B," respectively, in the tables of results below. They were obtained in the following sizes: One-eighth inch, five-thirty-seconds inch, three-sixteenths inch, and one-fourth inch (A electrodes five-sixteenths inch). It was planned to use the different sizes with the following currents: One-fourth inch—75, 110, and 145 amperes; five-thirty-seconds inch—145, 185, and 225 amperes; three-sixteenths inch—185, 225, and 260 amperes; one-fourth inch (five-sixteenths inch)—300 amperes. The electrodes were used both in the bare condition and after being slightly coated with a deoxidizing and refractory mixture. For coating, a paste of the following composition was used: Fifteen g graphite, 7.5 g magnesium, 4 g aluminum, 65 g magnesium oxide, 60 g calcium oxide. To this mixture was added 120 cm^3 of sodium silicate (40° Be) and 150 cm^3 of water. The electrodes were painted on one side only with

the paste. The quantity given above was found to be sufficient for coating 500 electrodes. The purpose of the coating was to prevent excessive oxidation of the metal of the electrode during fusion and to form also a thin protective coating of slag upon the fused metal.

Tension specimens only were prepared from the arc-fused metal; the modification and curtailment of the original program rendered this necessary. It is quite generally recognized that the tension test falls very short in completely defining the mechanical properties of any metal; it is believed, however, that the behavior of this material when stressed in tension is so characteristic that its general behavior under other conditions of stress, particularly when subjected to the so-called dynamic tests—that is—vibration and shock, can be safely predicted from the results obtained. In order to supplement the specimens made at this Bureau a series of six was also prepared by one of the large manufacturers of equipment for electric welding to be included in the investigation. These are designated as "C" in the tables below.

V. CHANGES IN COMPOSITION OF ELECTRODES AS A RESULT OF FUSION [4]

In Table 3 below are given the results of the analyses of several typical specimens of the two types of electrodes used for welding and also results of analyses of similar material after arc-fusion.

TABLE 3.—Results of Chemical Analyses of Electrodes Before and After Fusion [a]

Electrode		Carbon		Silicon		Manganese		Phosphorus	
Type	Diameter	Before	After	Before	After	Before	After	Before	After
	Inch	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
A	1/8	0.058	0.046 [b] 0.031	0.33	0.007 [b] 0.007	0.042	Tr Tr	0.002	0.005 [b] 0.005
A	5/32								
A	3/16	.022	.010 [b] .010 .033	.16	.012 [b] .014 .006	.038	Tr [b] Tr 0.069	.002	.003 [b] .002 .012
A	5/16		.050		.11		.014		.011
B	1/8	.15	.027 [b] .024	.06	.008 [b] .100	.47	Tr [b] Tr	.018	.002 [b] .004
B	5/32	.15		.001		.46		.014	
B	3/16								
B	1/4								
C	5/32								

[a] The electrodes which furnished the specimens used for analysis after fusion were not the identical ones used before fusion, but were the same stock.

[b] Results were obtained from the fusion of coated electrodes.

[4] Acknowledgment is due J. R. Cain, chemist, Bureau of Standards, for the method used in the determination of nitrogen. The details of the method, necessary precautions, and apparatus will be described in a separate publication.

TABLE 3.—Results of Chemical Analyses of Electrodes Before and After Fusion— Continued

Electrode		Sulphur		Copper [a]		Nitrogen [b]	
Type	Diameter	Before	After	Before	After	Before	After
	Inch	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
A	1/8	0.057	0.036 [c] .033			0.0030	0.156 .127 [c] .149 [c] .140
A	5/32					.0035	.140 .123 .124 [c] .121 [c] .119 [c] .113
A	3/16	.040	.033 [c] .035 .043		 0.058	.0040	.126 .131 .133 [c] .127 [c] .131 [c] .134
A	5/16		.026		.063	.0037	.117 [c] .111
B	1/8	.021	 .035 [c] .035			.0032	.152 .132 [c] .141 [c] .135
B	5/32	.017				.0035	.124 .121 .117 [c] .122 [d] .132 [c] .123
B	3/16					.0022 .0025	.119 .111 .112 [c] .106 [c] .108 [c] .094
B	1/4					.0014 .0022	
C	5/32						.133 .098

[a] Determinations for copper were not carried out upon the unfused electrodes.

[b] Each of the results reported in the "after" fusion columns, excepting [c], is the average of two determinations made on one separate specimen.

[c] Results were obtained from the fusion of coated electrodes.

[d] Average of nine determinations.

It will be noted that the general effect of the fusion is to render the two materials more nearly the same in composition. The loss of carbon and of silicon is very marked in each case where these elements exist in considerable amounts. A similar tendency may be noted for manganese. The coating with which the electrodes were covered appears to have had but little influence, if any, in preventing the oxidation of the carbon and other elements.

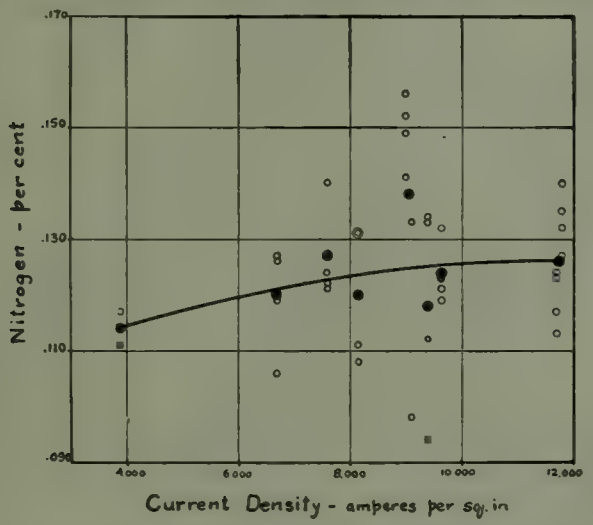


FIG. 4.—*Relation of current density to nitrogen content of the arc-fused steel*

The most noticeable change in composition is the increase in the nitrogen content of the metal. In general the increase was rather uniform for all specimens. In Table 4 are summarized the results of the nitrogen determinations together with the corresponding current density used for the fusion of the metal. In Fig. 4 the average nitrogen content found for the different conditions of fusion is plotted against the corresponding current density. Though no definite conclusion seems to be warranted, it may be said that, in general, the percentage of nitrogen taken up by the fused iron increases somewhat as the current density increases. With the lowest current densities used the amount of nitrogen was found to be appreciably less than for the higher current densities used. Paterson reports [3] an increase in nitrogen content under similar conditions.

TABLE 4.—Relation Between Nitrogen Content and Current Density

Diameter of electrode	Approximate current	Current density	Nitrogen content[a]			
			Specimen A	Specimen B	Specimen C	Average
	Amperes	Amp./in.²	Per cent	Per cent	Per cent	Per cent
1/8 inch	110	9 000	0.156 [b] 0.149	0.152 [b] 0.141		0.138
1/8 inch	145	11 800	.127 [b] .140	.132 [b] .135		.126
5/32 inch	145	7 600	.140 [b] .121	.124 [b] .122		.127
5/32 inch	185	9 650	.123 [b] .119	.121 [b,d] .132		.131
5/32 inch	225	11 700	.124 [b] .113	.117 [b] .123		[c]
5/32 inch	175	9 100			0.133 .098	[c]
3/16 inch	185	6 700	.126 [b] .127	.119 [b] .106		.120
3/16 inch	225	8 150	.131 [b] .131	.111 [b] .108		.120
3/16 inch	260	9 400	.133 [b] .134	.112 .094		.118
5/16 inch	300	3 900	.117 [b] .111			.114

[a] Average of two determinations.
[b] Coated electrodes.
[c] Included in average for C–D, 11 800.
[d] Average of nine determinations.
[e] Included in average for C–D, 9000.

VI. MECHANICAL PROPERTIES OF THE ARC-FUSED METAL

The mechanical properties of the two types of electrodes used as determined by the tension test are summarized in Table 5 below.

TABLE 5.—Tensile Properties of Electrodes

Electrode		Proportional limit	Ultimate tensile strength	Elongation in 2 inches	Reduction of area
Type	Diameter				
	Inch	Lbs./in.²	Lbs. in.²	Per cent	Per cent
A	5/32	39 000	65 800	16.5	69.2
A	3/16	48 000	62 100	9.0	69.3
A	5/16	34 000	60 100	14.0	66.4
A	5/16		57 300	15.5	67.6
B	5/32	67 000	88 600	4.5	51.3
B	3/16	58 500	84 700	7.0	59.8
B	1/4	37 500	66 300	15.0	61.4
B	1/4		67 900	15.5	62.4

In Table 6 are given the results of the mechanical tests made upon the tension specimens which were turned out of the blocks of metal resulting from the fusion of the electrodes. For purposes of comparison the chemical composition of the material is also included in the table. The specimens listed C–1, C–2, ...C–6 are the six which were prepared outside the Bureau and submitted for purposes of comparison. It was stated that they were prepared from bare electrodes five thirty-seconds inch in diameter, of type B, containing 0.17 per cent carbon and 0.5 per cent manganese.

TABLE 6.—Tensile Properties and Results of Chemical Analyses of 54 Specimens of Weld Metal (0.505 Inch Diameter Standard Tension Bar Used)

BARE ELECTRODES

Sample No.	Diameter of electrode	Direct current	Tensile properties			Elongation in 2 in.	Reduction of area	Hardness		C	Si	Mn	P	S	Cu	N_2
			Ultimate tensile strength	Yield point	Proportional limit			Brinell	Scleroscope							
	Inch	Amp.	Lbs./in.²	Lbs./in.²	Lbs./in.²	Per cent	Per cent									
A-2	1/8	110	49 850	36 600	25 000	6.0	6.5	108	17.0							0.156
A-3	1/8	145	51 950	36 250	30 000	8.0	13.0	114	18.0	0.046	0.007	Tr	0.005	0.036		.127
A-7	5/32	145	47 550			6.0	7.4	108	15.6							.140
A-8	5/32	185	48 100			8.0	8.7	104	16.2							.123
A-9	5/32	225	45 500			8.0	9.6	101	15.2							.124
A-4	3/16	185	50 600	33 750	29 500	5.5	13.5	105	16.2	.010	.012	Tr	.003	.033		.126
A-5	3/16	225	49 150	36 250	22 000	7.0	10.0	102	16.4	.033	.006	0.069	.012	.043	0.058	.131
A-6	3/16	260	50 950	33 750	28 800	10.5	12.0	107	17.4							.133
A-10	5/16	300	46 670			12.0	11.9	104	16.2	.050	.11	.014	.011	.026	.063	.117

COVERED ELECTRODES

AD-2	1/8	110	51 250	35 000	25 600	9.5	11.0	103	17.8							.149
AD-2[a]	1/8	110	43 000		23 000	5.0	9.0									
AD-3	1/8	145	51 100	33 750	25 000	8.5	10.5	110	17.2	.031	.007	Tr	.005	.033		.140
AD-3[a]	1/8	145	46 250		24 250	7.0	12.0									
AD-7	5/32	145	41 750			6.0	6.6	99	17.4							.121
AD-7[a]	5/32	145	46 950		25 500	8.0	9.4									
AD-8	5/32	185	44 620			6.5	5.8	103	16.8							.119
AD-8[a]	5/32	185	43 600		23 250	6.5	9.0									
AD-9	5/32	225	46 900			9.5	10.1	96	16.8							.113
AD-9[a]	5/32	225	41 550		25 500	5.0	6.5									
AD-4	3/16	185	51 200	35 000	30 000	10.5	10.5	101	17.2	.011	.014	Tr	.002	.035		.127
AD-4[a]	3/16	185	45 700		25 500	8.5	11.5									

AD-5	3/16	225	48 600	35 000	30 000	7.0	10.0	96	16.2							.131
AD-5[a]	3/16	225	46 250		23 750	11.5	12.0									
AD-6	3/16	260	47 500	34 500	31 500	9.0	9.0	97	14.6							.134
AD-6[a]	3/16	260	50 700			8.0	2.8	105	13.6							
AD-10	5/16	300	45 900			8.5	11.5	98	15.4							.111
BARE ELECTRODES																
B-2	1/8	110	52 650	37 000	37 000	7.5	7.5	114	18.8							[b].152
B-3	1/8	145	54 500	36 000	27 000	12.5	12.0	106	15.8	.027	.008	Tr	.002	.035		[b].132
B-4	5/32	145	46 450	33 500	26 000	5.0	7.0	102	16.2							[b].124
B-5	5/32	185	49 600	34 250	27 000	7.5	9.0	108	16.[illegible]							[b].121
B-6	5/32	225	49 500	30 500	28 000	9.0	7.5	110	16.6							[b].117
B-7	3/16	185	47 550		28 500	7.5	11.5	95	17.4							[b].119
B-8	5/32	225	42 900		18 750	7.5	16.2	101	16.2							[b].111
B-9	5/32	260	47 500		21 500	11.0	13.5	10[illegible]	15.4							[b].112
COVERED ELECTRODES																
BD-2	1/8	110	49 050	33 750	27 500	9.0	12.0	100	16.0							[b].141
BD-2[a]	1/8	110	44 400		20 000	6.5	9.4									
BD-3	1/8	145	52 100	34 300	30 500	12.5	16.0	116	16.0	.024	.010	Tr	.004	.035		[b].135
BD-3[a]	1/8	145	50 850		23 500	13.0	17.5									
BD-4	5/32	145	48 130	31 000	30 500	8.0	10.0	101	14.8							[b].122
BD-4[a]	5/32	145	41 750		21 000	6.0	9.5									
BD-5	5/32	185	49 086	31 730	29 000	12.5	13.0	97	16.0							[c].132
BD-5[a]	5/32	185	47 100		22 500	11.0	12.5									
BD-6	5/32	225	45 500	30 500	25 000	8.5	10.5	95	15.6							[b].123
BD-7	3/16	185	49 950		24 500	11.5	21.5	98	16.8							[b].106
BD-7[a]	3/16	185	51 150		33 750	14.5	19.5									
BD-8	3/16	225	41 500		17 850	6.0	12.7	99	13.8							[b].108
BD 8[a d]	[d] 3/16	[d] 225	48 750		21 250	12.5	16.0									
BD-9	3/16	260	46 350		24 000	10.0	15.0	99	14.6							[b].094

[a] Second test of duplicate specimen. [b] Average of two determinations. [c] Average of nine determinations. [d] Doubtful.

TABLE 6.—Tensile Properties and Results of Chemical Analyses of 54 Specimens of Weld Metal (0.505 Inch Diameter Standard Tension Bar Used)—Continued

BARE ELECTRODES

Sample No.	Diameter of electrode	Direct current	Tensile properties			Elongation in 2 in.	Reduction of area	Hardness		C	Si	Mn	P	S	Cu	N_2
			Ultimate tensile strength	Yield point	Proportional limit			Brinell	Scleroscope							
	Inch	Amp.	Lbs./in.²	Lbs./in.²	Lbs./in.²	Per cent	Per cent									
C-1	5/32	175	48 650	32 650	23 000	12.0	19.1									
C-2	5/32	175	45 200	32 400	23 000	7.5	16.6									
C-3	5/32	175	49 720	32 650	25 000	9.0	13.6									
C-4	5/32	175	54 500	32 500	25 000	11.0	17.5	118	19.2							[b] 0.133
C-5	5/32	175	50 900	32 500	24 000	15,0	23.0	109	15.2							[b] 0.098
C-6	5/32	175	50 500	33 500	23 000	12.0	16.0									

[b] Average of two determinations.

As an aid for more readily comparing the mechanical properties of the two types of arc-fused metal A and B, the results have been grouped as given in Table 7.

TABLE 7.—Tensile Properties and Hardness of Specimens of Weld Metal Prepared by the Bureau (Arranged in Order of Amperage Used)

Current used, amperes [a]	Tensile properties							
	Ultimate tensile strength				Yield point			
	Electrode A [b]		Electrode B [b]		Electrode A		Electrode B	
	Bare	Covered	Bare	Covered	Bare	Covered	Bare	Covered
	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²
110	49 850	51 250	52 650	49 050	36 600	35 000	37 000	33 750
	.....	[c] 43 000	.....	[c] 44 400	.....	.....	.....	.....
145 Series 1	51 950	51 000	54 500	52 100	36 250	36 750	36 000	34 300
	.....	[c] 46 250	.....	[c] 50 850	.....	.....	.....	.....
145 Series 2	47 550	41 750	46 450	48 130	.....	.....	33 500	31 000
	.....	[c] 46 950	.....	[c] 41 750	.....	.....	.....	.....
185 Series 1	48 100	44 620	49 600	49 086	.....	.....	34 250	31 730
	.....	[c] 43 600	.....	[c] 47 100	.....	.....	.....	.....
185 Series 2	50 600	51 200	47 550	49 950	33 750	35 000	.....	.....
	.....	[c] 45 700	.....	[c] 51 150	.....	.....	.....	.....
225 Series 1	45 500	46 900	49 500	45 500	.....	.....	30 500	30 500
	.....	[c] 41 550	.....	.....	.....	.....	.....	.....
225 Series 2	49 150	48 600	42 900	41 500	36 250	35 000	.....	.....
	.....	[c] 46 250	.....	.....	.....	.....	.....	.....
260	50 950	47 500	47 500	46 350	33 750	34 500	.....	.....
	.....	[c] 50 700	.....	.....	.....	.....	.....	.....
300	46 670	45 900	.....	.....	.....	.....	.....	.....
Average	48 900	46 600	48 800	47 450	35 300	34 650	34 250	32 250
	47 400		47 980		35 000		33 250	

[a] Size of electrode used: One-eighth inch diameter, 110 amperes and 145 amperes (series 1); 5/32 inch diameter, 145 amperes (series 2), 185 amperes (series 1), and 225 amperes (series 1); 3/16 inch diameter, 185 amperes (series 2), 225 amperes (series 2), and 260 amperes; 5/16 inch diameter, 300 amperes.

[b] A and B refer to the two types of electrodes used (Table 3).

[c] Duplicate specimen.

TABLE 7.—Tensile Properties and Hardness of Specimens of Weld Metal Prepared by the Bureau (Arranged in Order of Amperage Used)—Continued

Current used, amperes	Tensile properties—Continued							
	Elongation in 2 inches				Reduction in area			
	Electrode A		Electrode B		Electrode A		Electrode B	
	Bare	Covered	Bare	Covered	Bare	Covered	Bare	Covered
	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
110	6.0	9.5	7.5	9.0	6.5	11.0	7.5	12.0
		[a] 5.0		[a] 6.5		[a] 9.0		[a] 9.4
145, Series 1	8.0	8.5	12.5	12.5	13.0	10.5	12.0	16.0
		[a] 7.0		[a] 13.0		[a] 12.0		[a] 17.5
145, Series 2	6.0	6.0	5.0	8.0	7.4	6.6	7.0	10.0
		[a] 8.0		[a] 6.0		[a] 9.4		[a] 9.5
185, Series 1	8.0	6.5	7.5	12.5	8.7	5.8	9.0	13.0
		[a] 6.5		[a] 11.0		[a] 9.0		[a] 12.5
185, Series 2	5.5	10.5	7.5	11.5	23.5	10.5	11.5	21.5
		[a] 8.5		[a] 14.5		[a] 11.5		[a] 19.5
225, Series 1	8.0	9.5	9.0	8.5	9.6	10.1	7.5	10.5
		[a] 5.0				[a] 6.5		
225, Series 2	7.0	7.0	7.5	6.0	10.0	10.0	16.2	12.7
		[a] 11.5				[a] 12.0		
260	10.5	9.0	12.0	10.0	12.0	9.0	13.5	15.0
		[a] 8.0				[a] 2.8		
300	12.0	8.5			11.9	11.5		
Average	7.9	8.5	8.5	9.9	10.3	9.2	10.5	13.8
	7.9		9.0		9.6		12.5	

Current used, amperes	Brinell and scleroscope hardness							
	Electrode A				Electrode B			
	Brinell		Scleroscope		Brinell		Scleroscope	
	Bare	Covered	Bare	Covered	Bare	Covered	Bare	Covered
110	108	103	17.0	17.8	114	100	18.8	16.0
145, Series 1	114	110	18.0	17.2	106	116	15.8	16.0
145, Series 2	108	[illegible]	15.6	17.4	102	101	16.2	14.8
185, Series 1	104	103	16.2	16.8	[illegible]	97	16.2	16.0
185, Series 2	105	101	16.2	17.2	95	98	17.4	16.8
225, Series 1	101	[illegible]	15.2	16.8	110	95	16.6	15.6
225, Series 2	102	[illegible]	16.4	16.2	101	[illegible]	16.2	13.8
260	107	97	17.4	14.6	102	[illegible]	15.4	14.6
300	104		16.2					
Average	106	100	16.5	16.8	105	101	16.6	15.5
	103		16.6		103		16.0	

[a] Duplicate specimen.

The characteristic appearance of specimens after testing, illustrating their behavior when stressed in tension until rupture occurs, is shown in Figs. 5, *a* and *b*. These represent two views

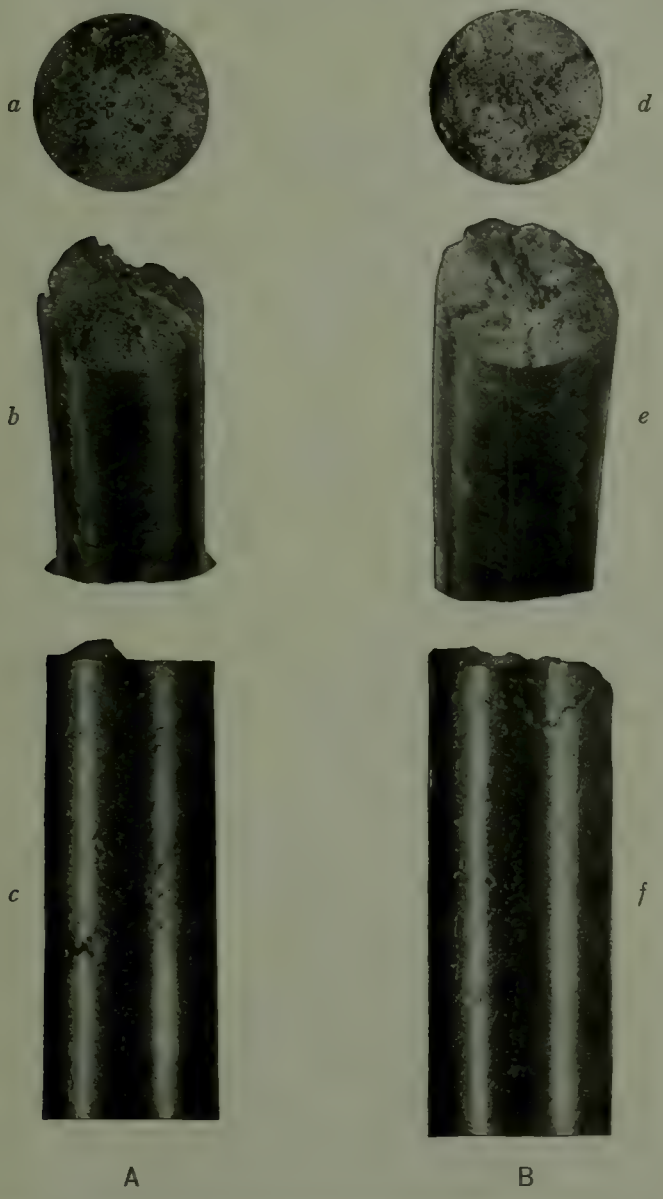


FIG. 5.—*Characteristic appearance of tension specimens after test.* × 2

A, specimen B-3 (Table 6); *B*, specimen BD-6 (Table 6); *a*, *d*, face of fracture, viewed normally; *b*, *e*, fractured end of specimen, viewed at an angle of 45°; *c*, *f*, side view of specimen

of the face of the fracture, one in which the line of vision is perpendicular to the face, the other at an angle of 45°, together with a side view of the cylindrical surface of the specimen. The features shown in Fig. 5 are characteristic of all the specimens tested, though in some they were much more pronounced than those shown. The fracture of the specimen in all cases reveals interior flaws. In some of the specimens, however, these are microscopic and of the character shown in Fig. 12. Although many of the specimens (from the results of Table 6) appear to have a considerable elongation, it is seen from Fig. 5 that the measured elongation does not truly represent a property of the metal itself. It is due rather to interior defects which indicate lack of perfect union of succeeding additions of metal during the process of fusion. The surface markings of the specimen after stressing to rupture are very similar to those seen in the familiar "flaky steel."

It appears from the results above that, as far as the mechanical properties are concerned, nothing was gained by coating the electrodes. The results show no decided superiority for either of the two types of electrodes used. This may be expected, however, when one considers that the two are rendered practically the same in composition during fusion by the burning out of the carbon and other elements.

The results of the tension tests upon the C series of specimens, which were made outside of this Bureau and submitted to be included in the investigation, show no marked difference between these samples and those prepared by this Bureau. In all cases the results obtained in the tension test are determined by the soundness of the metal and do not necessarily indicate the real mechanical properties of the material.

The results of the hardness determinations do not appear to have any particular or unusual significance. The variations are of the same general nature and relative magnitude as the variations observed in the results of the tension test. In general, the higher hardness number accompanies the higher tensile values, though this was not invariably so. As previously noted, specimens were prepared for the purpose of showing the relation between the direction in which the stress is applied and the manner of deposition of the metal. The metal was deposited in the form shown in Fig. 3, except that the "beads" extended across the piece rather than lengthwise, hence the "beads" of fused metal were at right angles to the direction in which the tensional stress

was applied. The results of the tension tests show that these two specimens (AW–1 and AW–2) were decidedly inferior to those prepared in the other manner, as shown in the table below.

TABLE 8.—Mechanical Properties of Arc-Fused Metal Deposited at Right Angles to Length of Specimen

Specimen	Proportional limit	Ultimate tensile strength	Elongation in 2 inches	Reduction of area
	Lbs./in.²	Lbs./in.²	Per cent	Per cent
AW–1	22 500	40 450	6.5	8.5
AW–2	22 500	39 500	4.0	3.0

VII. STRUCTURAL FEATURES: METALLOGRAPHIC EXAMINATION

1. MACROSTRUCTURE

The general condition of the metal resulting from the arc fusion is shown in Figs. 6 and 7, which show longitudinal medial sections of a series of the tension bars adjacent to the fractured end. The metal in all of these specimens was found to contain a considerable number of cavities and oxide inclusions; these are best seen after the surfaces are etched with a 10 per cent aqueous solution of copper-ammonium chloride. In many of the specimens the successive additions of metal are outlined by a series of very fine inclusions (probably oxide) which are revealed by the etching. There appears to be no definite relation between the soundness of the metal and the conditions of deposition—that is, for the range of current density used—nor does either type of electrode used show any decided superiority over the other with respect to porosity of the resulting fusion. In Fig. 8, *a* and *b*, is shown the appearance of a cross section of one of the blocks of arc-fused metal prepared outside of this Bureau by skilled welding operators. The condition of this material is quite similar to that prepared by this Bureau. The appearance of the fracture of one of the tension specimens made from such material is shown in Fig. 8*c*. The same defects as may be seen in the cross sections (Fig. 8, *a* and *b*) are also to be found on the fractured end of the bar; in addition to these, there appears to be a seam of unwelded metal.

The later microscopic study of the material also revealed further evidence of unsoundness in all the three types, A, B, and C.

FIG. 6.—*Macrostructure of arc-fused metal from electrodes of type A.* × 2

Medial longitudinal sections of tension specimens (Table 6) were etched with 10 per cent aqueous solution of copper ammonium chloride. *a*, specimen AD–6, $\frac{1}{8}$-inch covered electrode, 260 amperes; *b*, specimen A–5, $\frac{1}{16}$-inch bare electrode, 225 amperes; *c*, specimen A–6, $\frac{1}{16}$-inch bare electrode, 260 amperes; *d*, specimen A–3, $\frac{1}{8}$-inch bare electrode, 145 amperes; *e*, specimen A–4, $\frac{1}{16}$-inch bare electrode, 185 amperes; *f*, specimen AD–2, $\frac{1}{8}$-inch covered electrode, 110 amperes

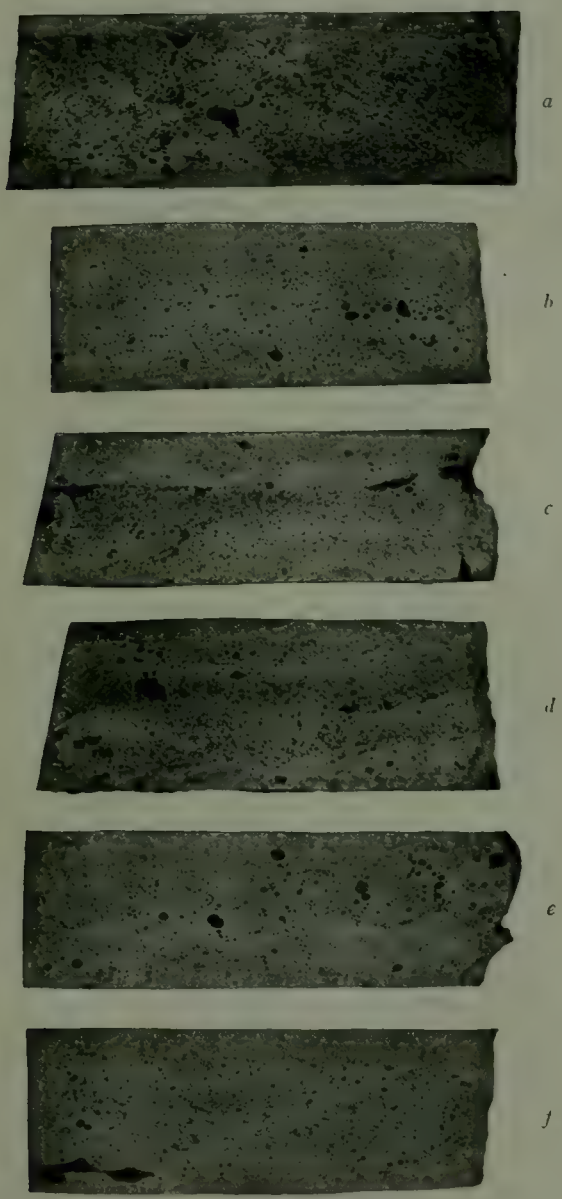


FIG. 7.—*Macrostructure of arc-fused metal from electrodes of type B.* × 2

Medial longitudinal sections of tension specimens (Table 6) were etched with 10 per cent aqueous solution of copper ammonium chloride. *a*, specimen B–4, $\frac{5}{32}$-inch bare electrode, 145 amperes; *b*, specimen B–5, $\frac{3}{16}$-inch bare electrode, 185 amperes; *c*, specimen B–3, $\frac{1}{8}$-inch bare electrode, 110 amperes; *d*, specimen B–1, $\frac{1}{8}$-inch bare electrode, 145 amperes; *e*, specimen BD–6, $\frac{5}{32}$-inch covered electrode, 225 amperes; *f*, specimen BD–4, $\frac{5}{32}$-inch covered electrode, 145 amperes

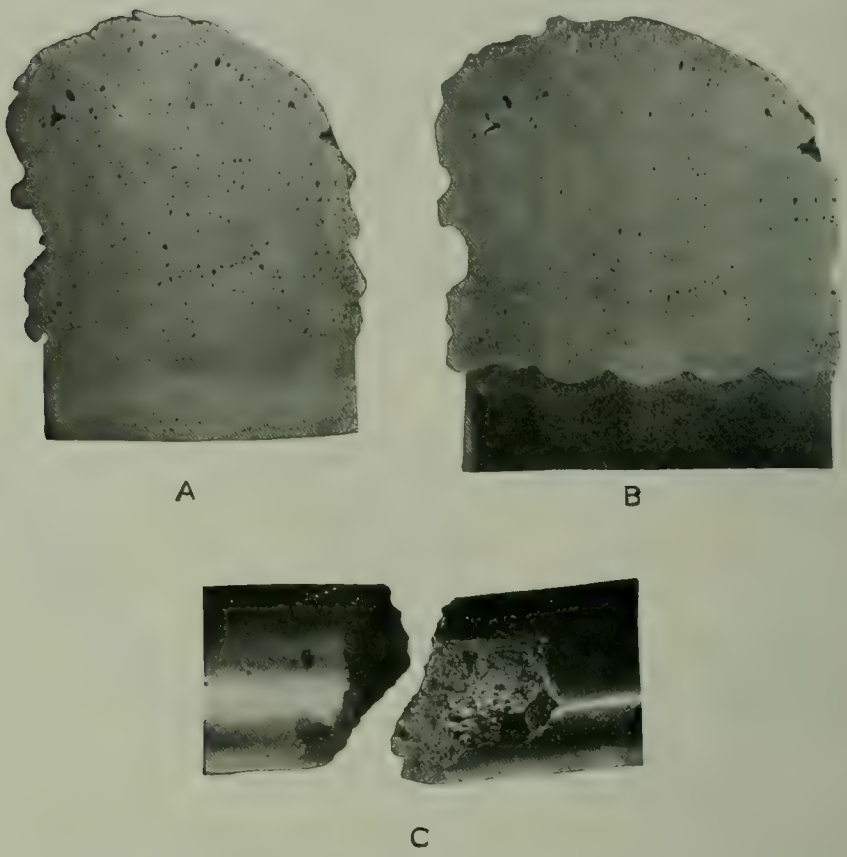


FIG. 8.—*Macrostructure of arc-fused metal, Type C*

A, cross section of the block of arc-fused metal from which specimen C–1 (Table 6) was turned; unetched, ×1. *B*, same as *A*, etched with 10 per cent aqueous solution of copper ammonium chloride, ×1.2; *C*, appearance of fractured tension specimen C–4 (Table 6), ×2

2. MICROSTRUCTURE

(*a*) GENERAL FEATURES.—For purposes of comparison the microstructure of the electrodes before fusion is shown in FIG. 9. The A electrodes have the appearance of steel of a very low carbon content. In some cases they were in the cold-rolled state; all showed a considerable number of inclusions. The B electrodes have the structure of a mild steel and are much freer from inclusions than are those of the other type. It is undoubtedly true, however, that the condition of the arc-fused metal with respect to the number of inclusions is a result of the fusion rather than of the initial state of the metal.

It is to be expected that the microstructure of the material after fusion will be very considerably changed, since the metal

is then essentially the same as a casting. It has some features, however, which are not to be found in steel as ordinarily cast.

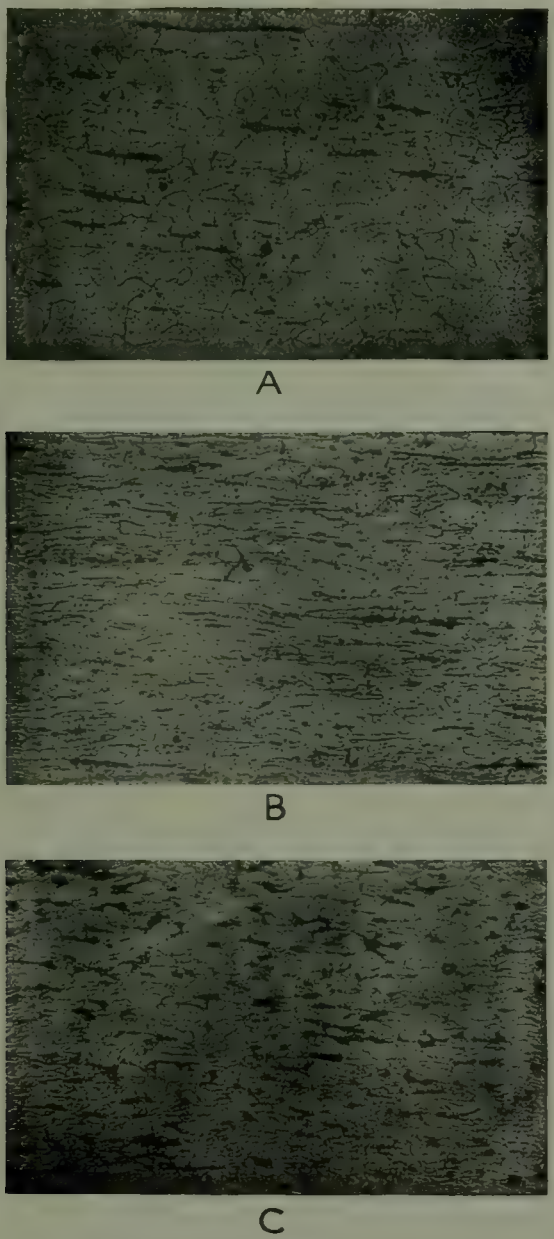


FIG. 9.—*Microstructure of electrodes before fusion.* × 100

A, electrode of type A, $\frac{5}{32}$ inch, annealed as received; *B*, electrode of type B, $\frac{1}{8}$ inch, cold drawn as received; *C*, electrode of type B, $\frac{1}{8}$ inch, annealed as received. Etching reagent, 5 per cent alcoholic solution of picric acid

The general type of microstructure was found to vary in the different specimens and to range from a condition which will be designated as "columnar" to that of a uniform fine equi-axed

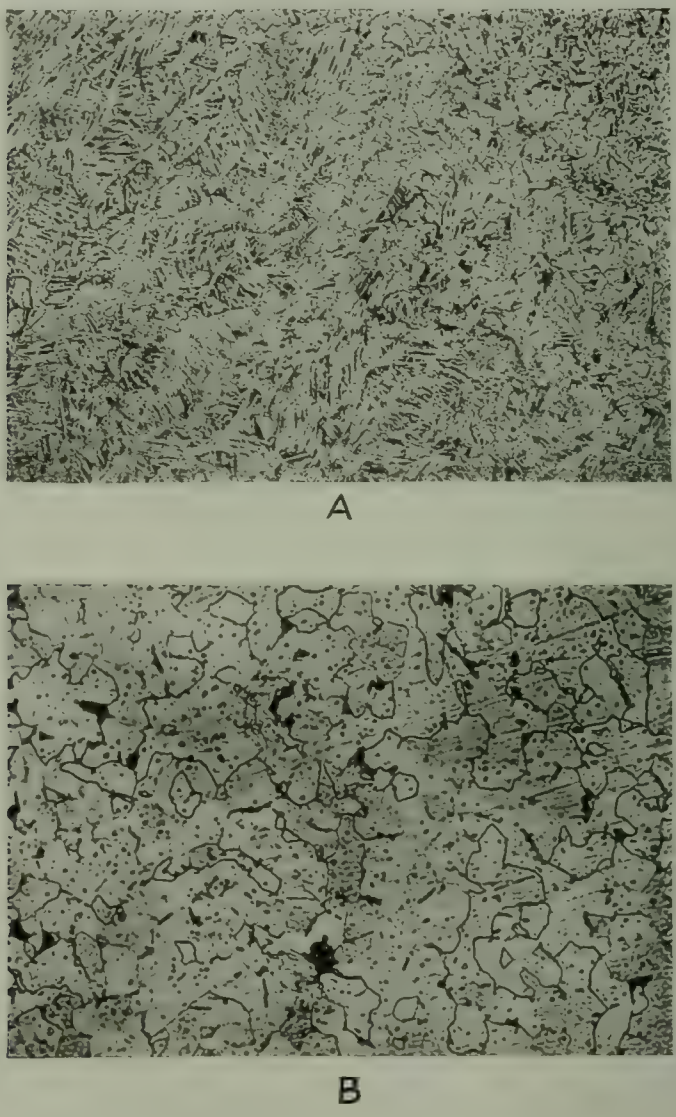


FIG. 10.—*Typical microstructures of arc-fused metal, designated as "columnar" and "equiaxed."* × 100

A, columnar condition (specimen B–2, Table 6); etching reagents, 5 per cent alcoholic solution of picric acid; an equiaxed condition is shown in the upper right-hand corner; *B*, equiaxed condition (specimen AD–3, Table 6). Etching reagent, 2 per cent alcoholic solution of nitric acid

crystalline arrangement. This observation held true for both types of electrodes, whether bare or covered. Fig. 10 shows the

two extremes of microstructure which were found. In the examination of cross sections of the blocks of arc-fused metal, it was noticed that the equi-axed type of structure is prevalent throughout the interior of the piece and the columnar is to be found generally nearer the surface; that is, in the metal deposited last. It may be inferred from this that the metal of the layers which were deposited during the early part of the preparation of the specimen is refined considerably by the successive heatings to which it is subjected as additional layers of metal are deposited.

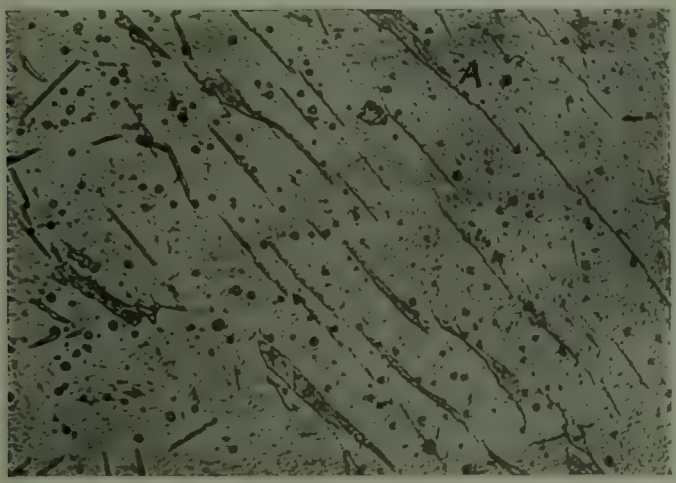


FIG. 11.—*Microstructure of arc-fused steel showing characteristic features, pearlite islands, "needles," and oxide inclusions.* × 750

The specimen (AD–2, Table 6) was etched with 2 per cent alcoholic solution of nitric acid

The general type of structure of the tension bars cut from the blocks of arc-fused metal will vary considerably according to the amount of refining which has taken place as well as the relative position of the tension specimen within the block. In addition it was noticed that the columnar and coarse equi-axed crystalline condition appears to predominate with fusion at high current densities.

(*b*) MICROSCOPIC EVIDENCE OF UNSOUNDNESS.—In all of the specimens of arc-fused metal examined microscopically there appear to be numerous tiny globules, apparently oxide, as shown in Figs. 10, 11, 12, and 13. A magnification of 500 diameters is usually necessary to show these inclusions. In general, they appear to have no definite arrangement but occur indiscriminately throughout the crystals of iron.

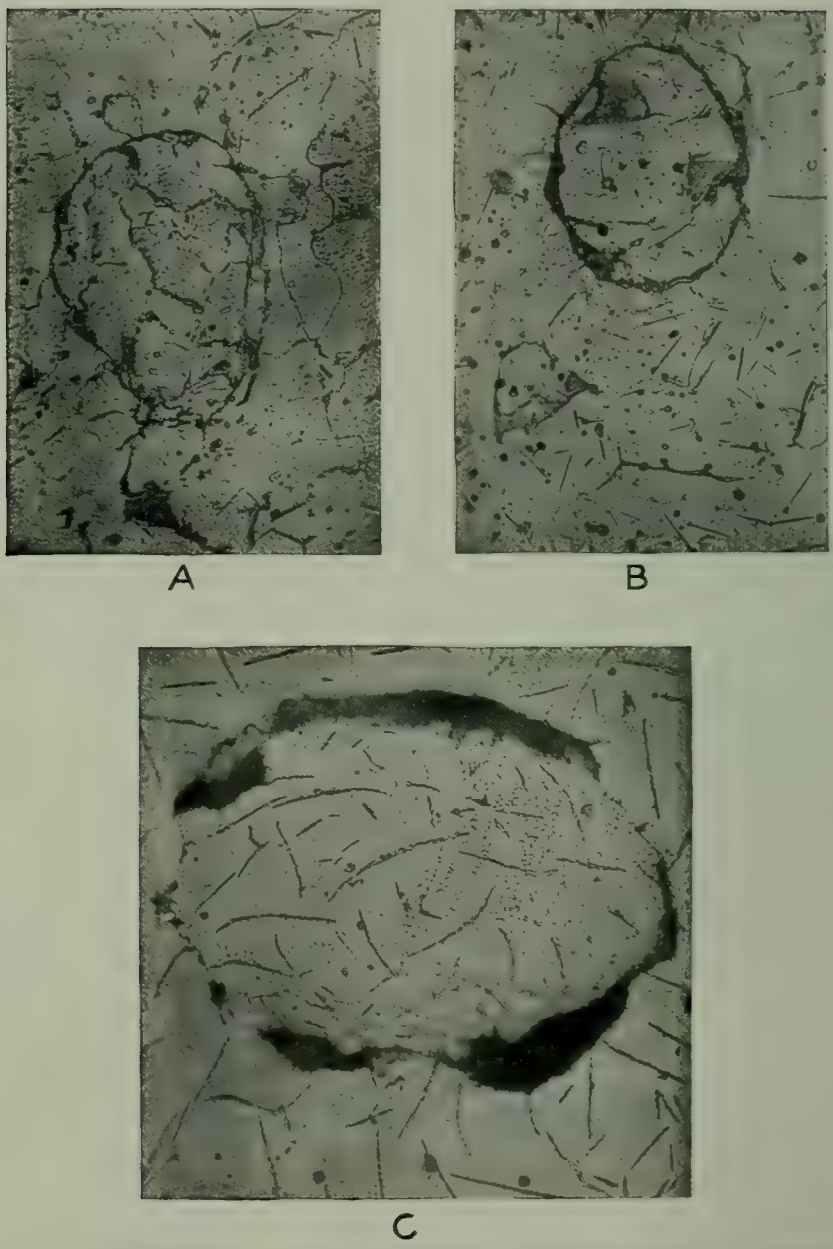


FIG. 12.—*Microstructure of arc-fused steel showing characteristic feature of "metallic-globule" inclusions.* × 500

A, specimen AD–9 (Table 6); *B*, specimen AD–10 (Table 6); *C*, specimen A–10 (Table 6) after heating 6 hours in vacuo. Etching reagent, 2 per cent alcoholic solution of nitric acid

A type of unsoundness frequently found is that shown in Fig. 12; this will be referred to as "metallic-globule inclusions." In

general, these globules possess a microstructure similar to that of the surrounding metal, but are enveloped by a film, presumably of oxide. It seems probable that they are small metallic particles which were formed as a sort of spray at the tip of the electrode and which were deposited on the solidified crust surrounding the pool of molten metal directly under the arc. These solidified particles apparently are not fused in with the metal which is subsequently deposited over them; that is, during the formation of this same layer and before any brushing of the surface occurs. By taking extreme precautions during the fusion a great deal of the unsoundness may be avoided, and the mechanical properties of the metal may be considerably improved. However, the specimens as described are more representative of actual present practice in welding.

(*c*) CHARACTERISTIC "NEEDLES" OR "PLATES."—The most characteristic feature of the steel after fusion is the presence of numerous lines or needles within the crystals. The general appearance of this feature of the structure is shown in Fig. 13. The number and the distribution of these "needles" were found to vary greatly in the different specimens. They are most abundant in the columnar and in the coarse equi-axed crystals. The finer equi-axed crystals in some specimens were found to be quite free from them, although exceptions were found to this rule. In general, a needle lies entirely within the bounds of an individual crystal. Some instances were found, however, where a needle appears to lie across the boundary and so lies within two adjacent crystals. Several instances of this tendency have been noted in the literature on this subject ([12]—Fig. 8; [27]—Fig. 30; [18]—p. 251, Fig. 14 B). The needles have an appreciable width, and, when the specimen is etched with 2 per cent alcoholic nitric acid, they appear much the same as cementite; that is, they remain uncolored, although they may appear to widen and darken if the etching is prolonged considerably. The apparent widening is evidently due to the attack of the adjacent ferrite along the boundary line between the two. The tendency of the needles to darken when etched with a hot alkaline solution of sodium picrate as reported by Comstock [12] was confirmed. Fig. 13, *c* illustrates the appearance when etched in this manner. The needles are sometimes found in a rectangular grouping; that is, they form angles of 90° with one another. In other cases they appear to be arranged along the octahedral planes of the crystal;

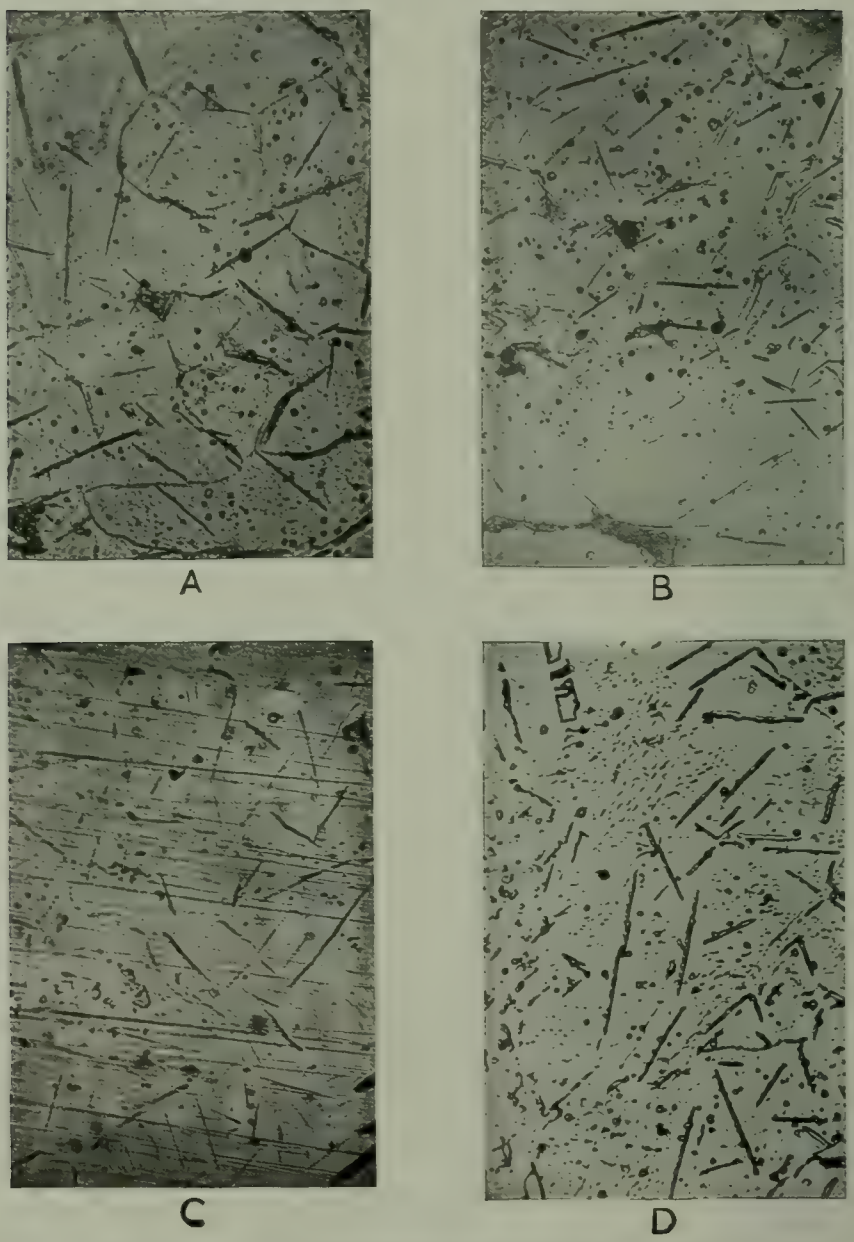


FIG. 13.—*Microstructure of arc-fused steel showing characteristic "needles" or "plates."*
× 500

A, specimen BD (Table 6); *B*, specimen BD-3 (Table 6); *C*, specimen A-5 (Table 6) (the material was used for thermal analysis (Sec. VII, 4) and was heated to approximately 900 ° C four times and cooled in vacuo); *D*, specimen of welded ship plate, one of the specimens used in the preliminary examinations (sec. 3); in addition to the usual needles a second generation of very small ones may be seen. Etching reagents. *A* and *B*, 5 per cent alcoholic solution of picric acid; *C*, hot alkaline solution of sodium picrate; *D*, 2 per cent alcoholic solution of nitric acid

that is, at 60° to one another. This is best seen in specimens which have been heated as explained in section 3 below.

In some of the specimens certain crystals showed groups of very fine short needles (Fig. 13*d*). The needles comprising any one group or family are usually arranged parallel to one another, but the various groups are often arranged definitely with respect to one another in the same manner as described above. Similar needles have been reported in the literature by Miller [**14**].

An attempt was made [5] to determine whether the so-called lines or needles were really of the shape of needles or of tiny plates or scales. An area was carefully located on a specimen prepared for microscopic examination, which was then ground down slightly and repolished several times. It was possible to measure the amount of metal removed during the slight grinding by observing the gradual disappearance of certain of the spherical oxide inclusions the diameter of which could be accurately measured. By slightly etching the specimen after polishing anew it was possible to follow the gradual disappearance of some of the most prominent needles and to measure the maximum "depth" of such needles. It was concluded from the series of examinations that the term "plate" is more correctly descriptive of this feature of the structure than "line" or "needle." The thickness of the plate—that is, the width of the needle—varies from 0.0005 to 0.001 mm, and the width of the plate ("depth") may be as great as 0.005 mm. The persistence of the plates after a regrinding of the surface used for microscopical examination may be noted in some of the micrographs given by Miller [**21**]. The authors are not aware, however, of any other attempt to determine the shape of these plates by actual measurements of their dimensions.

The usual explanation of the nature of these plates is that they are due to nitrogen which is taken up by the iron during its fusion. Other suggestions which have been offered previously attribute them to oxide of iron and to carbide. The suggestion concerning oxide may be dismissed with a few words. The plates are distinctly different from oxide in their form and their behavior upon heating. It is shown later (sec. 3) that the tiny oxide globules coalesce into larger ones upon prolonged heating in vacuo, the plates also increase in size and become much more distinct (Fig. 25, *b*, *d*, *f*). In no case, however, was any intermediate stage

[5] This was carried out by P. D. Merica, formerly physicist of the Bureau of Standards.

between the globular form and the plate produced such as would be expected if both were of the same chemical nature.

A

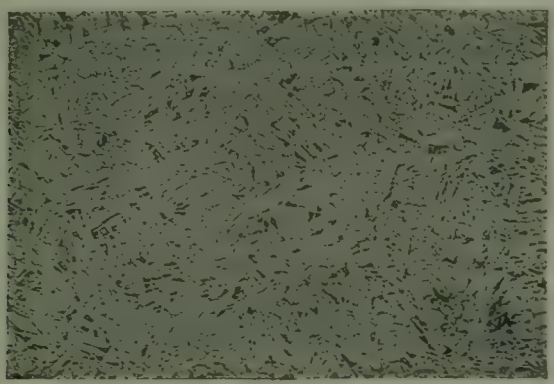
B

FIG. 14.—*Characteristic microstructure of "nitrogenized" iron.* × 500

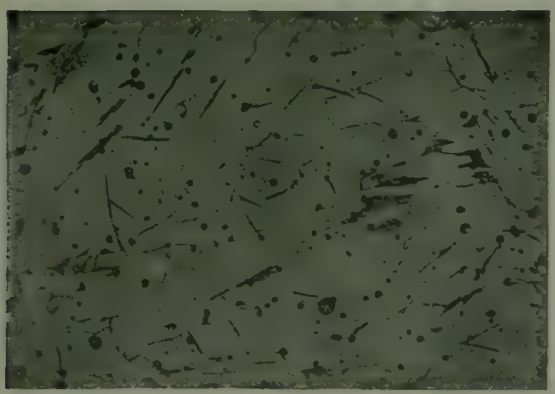
Electrolytic iron was heated for some hours at approximately 650° C in ammonia gas. It takes up nitrogen readily under these conditions. *A*, two types of needles occur, etching reagent, 2 per cent alcoholic nitric acid; *B*, same specimen as *A* (another area). Etching reagent, hot alkaline solution of sodium picrate

Regarding the assumption that they are cementite plates, it may be said that the tendency during fusion is for the carbon to be burnt out, thus leaving an iron of low carbon content. In all the

specimens islands of pearlite (usually with cementite borders) are to be found and may easily be distinguished with certainty. The number of such islands in any specimen appears to be sufficient to



A



B

FIG. 15.—*Microstructure of arc-fused steel produced in an atmosphere of carbon dioxide*

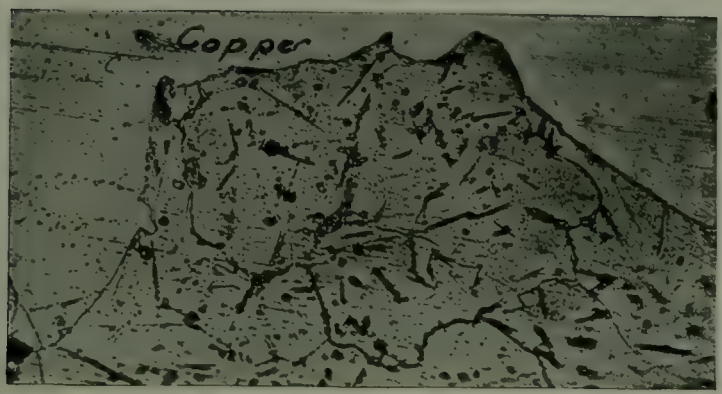
The structure is quite similar to that of the same fused in air; $\frac{1}{8}$-inch electrode of type A was used with 150 amperes. Etching reagent, 5 per cent alcoholic solution of picric acid: magnification, *A*, × 100; *B*, × 500

account for the carbon content of the material as revealed by chemical analysis. In some cases the pearlite islands are associated with a certain type of lines or needles such as are shown in Fig. 11. These needles, however, appear distinctly different from those of the prevailing type and are usually easily distinguished from them. The thermal analysis of arc-fused iron (Sec. VII, 4)

gives further evidence that the plates are not to be attributed to carbon.

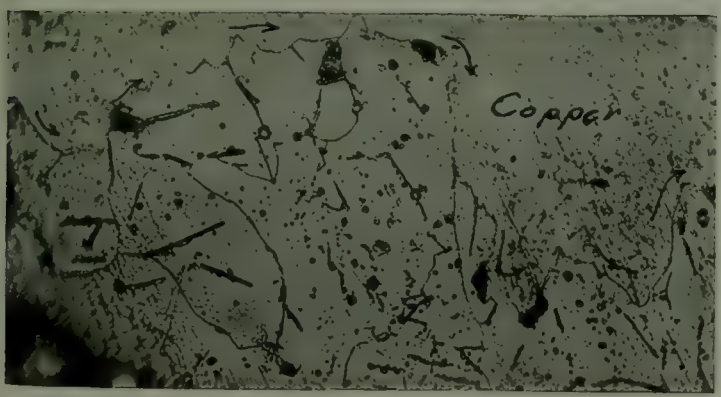
The fact that the plates found in the arc-fused metal are identical in appearance and in behavior (for example, etching) as those found in iron which has been nitrogenized is strong evidence that both are of the same nature. Fig. 14*a* shows the appearance of the plates produced in electrolytic iron by heating it for some time in pure ammonia gas. These plates behave in the same characteristic manner when etched with sodium picrate as do those occurring in arc-fused iron (Fig. 14*b*). The fact that the nitrogen content of the steel is increased by the arc fusion also supports the view that the change which occurs in the structure is due to the nitrogen. The statement has been made by Ruder [24] that metal fused in the absence of nitrogen—that is, in an atmosphere of carbon dioxide or of hydrogen—does not contain any plates, and hence the view that the plates are due to the nitrogen is very much strengthened. In Fig. 15 the appearance of specimens prepared at this Bureau by arc fusion of electrodes of type A in an atmosphere of carbon dioxide is shown. The microscopic examination of the fused metal shows unmistakable evidence of the presence of some plates, although they differ somewhat from those found in nitrogenized iron and in metal fused in the air by the electric arc. Evidently they are due to a different cause from the majority of those formed in the iron fused in air. For convenience, in the remainder of the discussion the plates in the arc-fused metal will be referred to as "nitride plates."

(*d*) Relation of Microstructure to the Path of Rupture.—The faces of the fracture of several of the tension specimens after testing were heavily plated electrolytically with copper, so as to preserve the edges of the specimens during the polishing of the section, and examined microscopically to see if the course of the path of rupture had been influenced to an appreciable extent by the microstructural features. In general, the fracture appears to be intercrystalline in type. Along the path of rupture in all of the specimens were smooth-edged hollows, many of which had evidently been occupied by the "metallic globules" referred to above, while others were gas holes or pores. Portions of the fracture were intracrystalline and presented a jagged outline, but it can not be stated with certainty whether the needles have influenced the break at such points or not. Fig. 16 shows the appearance of some of the fractures and illus-



A

B



C

FIG. 16.—*Microstructure of arc-fused steel showing the relation of the path of rupture produced in tension to structural features.* × 500

A, Specimen B-4 (Table 6); *B*, same specimen as *A*, another spot; *C*, specimen B-6 (Table 6). The layer of copper indicated was deposited electrolytically on the fractured face of the specimen to protect it during polishing. Etching reagent, 2 per cent alcoholic solution of nitric acid

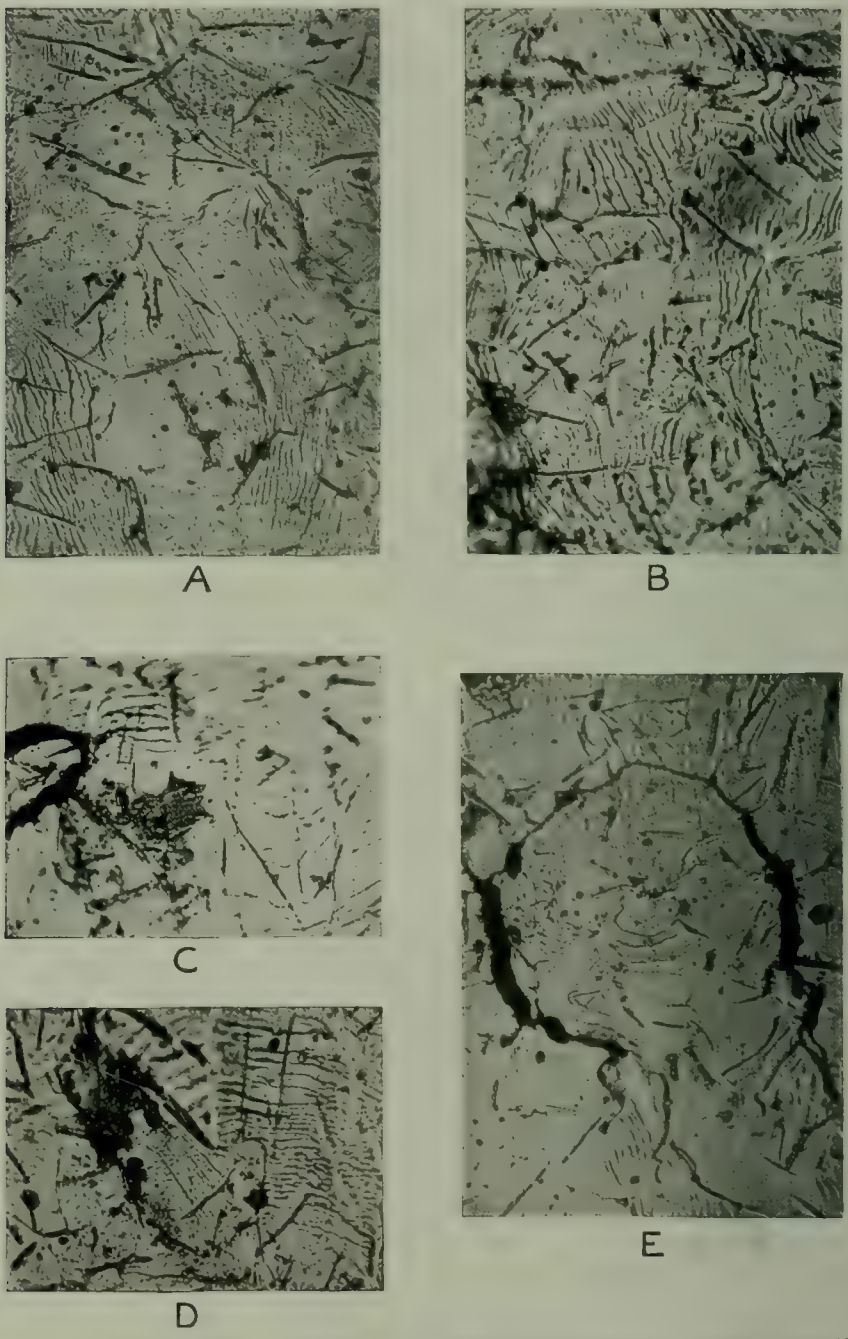


FIG. 17.—*Microstructure of arc-fused iron showing the behavior of "nitride plates" during plastic deformation of the metal.* × 500

A thin slice of specimen BD–2 (Table 6) was bent after polishing and etching with 2 per cent alcoholic nitric acid through an angle of 20° (approximate). *A* and *B* show that the "plates" have an effect upon the course of the slip bands similar to that of grain boundaries; that is, they cause a change of their direction; *C* and *D* show that a "faulting" of the "plates" sometimes occurs in grains which are very severely distorted; *E* shows that fracture upon bending usually starts in the oxide film inclosing "metallic globules" or in similar unsound areas

trates that, in general, the nitride plates do not appear to determine to any appreciable extent the course of the path of rupture.

The behavior of the plates under deformation can best be seen in thin specimens of the metal which were bent through a considerable angle. Results of examination of welds treated in this manner have been described by Miller [**14**]. Small rectangular plates of the arc-fused metal, approximately three thirty-seconds of an inch thick, were polished and etched for microscopic examination and were then bent in the vise through an angle of 20° (approximate).

In Fig. 17 are given micrographs illustrating the characteristic behavior of the material when subjected to bending. For moderate distortion the nitride plates influence the course of the slip bands in much the same way that grain boundaries do; that is, the slip bands terminate usually on meeting one of the plates with a change of direction so that they form a sharper angle with the plate than does the portion of the slip band which is at some distance away. When the deformation is greater, the slip bands occur on both sides of the nitride plate, but usually show a slight variation in direction on the two sides of the nitride plate; this is often quite pronounced at the point where the plate is crossed by the slip band. In a few cases evidence of the "faulting" of the plate as a result of severe distortion was noted. This was rare, however, because of the nature of the metal. On account of the inclusions and other features of unsoundness of the metal, rupture occurs at such points before the sound crystals have been sufficiently strained to show the characteristic behavior of the plates. Fig. 17*e* shows the beginning of a fracture around one of the metallic globule inclusions before the surrounding metal has been very severely strained. For this reason the influence of the plates on the mechanical properties of the crystals can not be stated with certainty. It would appear, however, that on account of the apparently unavoidable unsoundness of the metal, any possible influence of the nitride plates upon the mechanical properties of the material is quite negligible.

Some of the same specimens used for cold bending were torn partially in two after localizing the tear by means of a saw-cut in the edge of the plate. The specimen was then copper plated and prepared for microscopic examination, the surface having been ground away sufficiently to reveal the weld metal with the tear in it. Fig. 18 shows the appearance of the extreme end of a tear produced in this manner. The nitride plates do not appear to

have determined to any extent the path taken in the rupture produced in this manner.

3. EFFECT OF HEAT TREATMENT UPON STRUCTURE

With the view of possibly gaining further information as to the nature of the plates (assumed to be nitride) which constitute such a characteristic feature of the microstructure, a series of heat treatments was carried out upon several specimens of arc-fused electrodes of both types. Briefly stated, the treatment consisted in quenching the specimens in cold water after heating them for a period of 10 or 15 minutes at a temperature considerably above that of the Ac_3 transformation; 925°, 950°, and 1000° C were the temperatures used. After microscopical examination of the different quenched specimens they were tempered at different temperatures, which varied from 650 to 925° C for periods of 10 to 20 minutes. The samples which were used were rather small in size, being only one-eighth of an inch thick in order that the effect of the treatment should be very thorough,

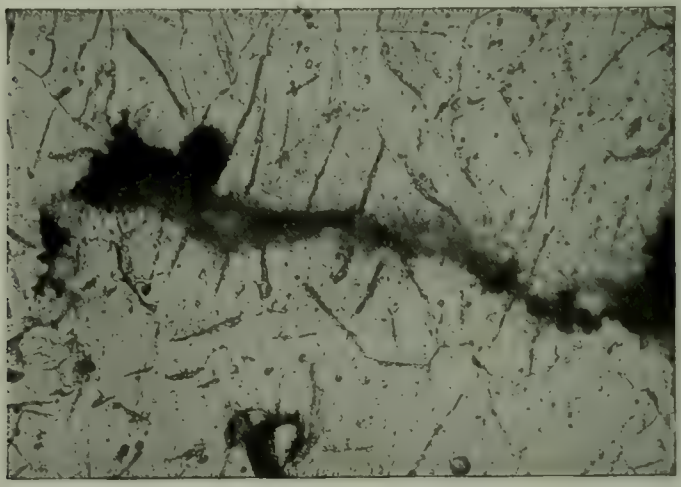


FIG. 18.—*Microstructure of arc-fused steel, showing the relation of the path of rupture to the "nitride plates."* × 500

A thin plate of metal (specimen of Fig. 17) was slowly torn after being partially sawed in two. The tear bears no evident relationship to the plates that lie in its path. Etching reagent used before fracturing the metal, 2 per cent alcoholic solution of nitric acid

and were taken from test bars A–2, A–6, AD–10, B–2, B–6, and B–9. These represented metal which had been deposited under different conditions of current density as shown in Table 6. No plates were found to be present in any of the specimens after quenching. Fig. 19 shows the appearance of one of the quenched

bars, which condition is typical of all. The structure indicates that the material comprising the plates had dissolved in the matrix of iron and had been retained in this condition upon quenching. The needlelike striations within the individual grains are characteristic of the condition resulting from the severe quenching and are to be observed at times in steel of a very low carbon content. Fig. 20 shows the appearance of one of the A electrodes (five thirty-seconds of an inch) quenched in cold water from 1000° C. Some of the crystals of the quenched iron also show interior markings somewhat similar in appearance to the nitride plates (Fig. 20*b*). These are, however, probably of the same nature as the interior treelike network sometimes seen in ferrite which has been heated to a high temperature.[6]

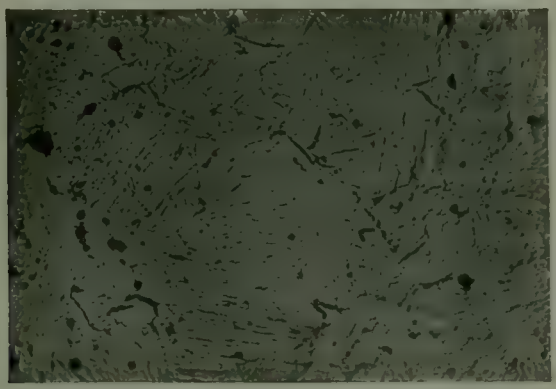


FIG. 19.—*Microstructure of arc-fused steel after quenching in water from 1000° C.* × 500

The plates characteristic of the material (specimen AD-10) before heating have disappeared and a martensitic pattern has been produced. Etching reagent, 5 per cent alcoholic solution of picric acid

The striations were found to be most pronounced in the specimens of arc-fused metal which were quenched from the highest temperatures, as might be expected. Braune [**49**] states that nitride of iron in quenched metal is retained in solution in the martensite. The same may be inferred from the statement by Giesen [**43**] "that in hardened steel, it (nitrogen) occurs in martensite." Ruder [**28**] has also shown that nitrogenized electrolytic iron (three hours at 700° C in ammonia), after being water quenched from a temperature of 600 to 950° C, shows none of the plates which were present before the specimen was heated.

[6] H. S. Rawdon and H. Scott, B. S. Sci. Papers, No. 356, Fig. 2*b*.

The sets of specimens (A–2, A–6, AD–10, B–2, B–6, and B–9) quenched from above the temperature of the Ac_3 transformation were heated to various temperatures, 650, 700, 800, and 925° C.

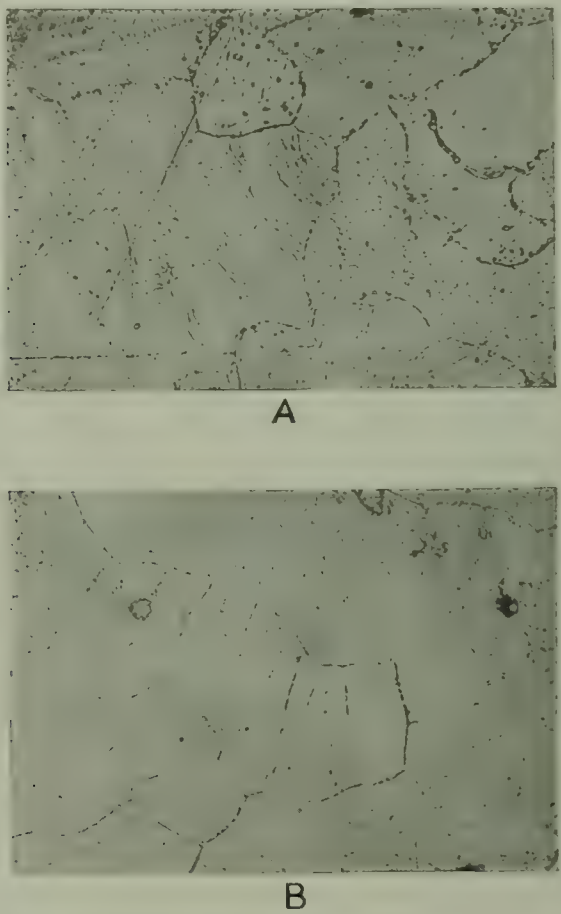


FIG. 20.—*Microstructure of "carbonless" steel after quenching in water from 1000° C.* × 500

A sample of electrode metal, type A, $\frac{1}{8}$ inch diameter, was used, being maintained 10 minutes at temperature before quenching. *A*, Some of the ferrite crystals (particularly near the surface of the specimen) show a faint pattern similar to that of Fig. 19; *B*, other ferrite crystals show interior markings somewhat suggestive of the plates of the arc-fused metal. Etching reagent, 2 per cent alcoholic solution of nitric acid

In all cases the specimens were maintained at the maximum temperature for approximately 10 to 15 minutes and then cooled in the furnace. Figs. 21 to 24, inclusive, summarize the resulting effects upon the structure. Heating to 650° C is not sufficient to allow the plates to redevelop, but in the specimens heated to 700° C a few small ones were found. The effect is progressively more pronounced with the increased temperature of tempering,

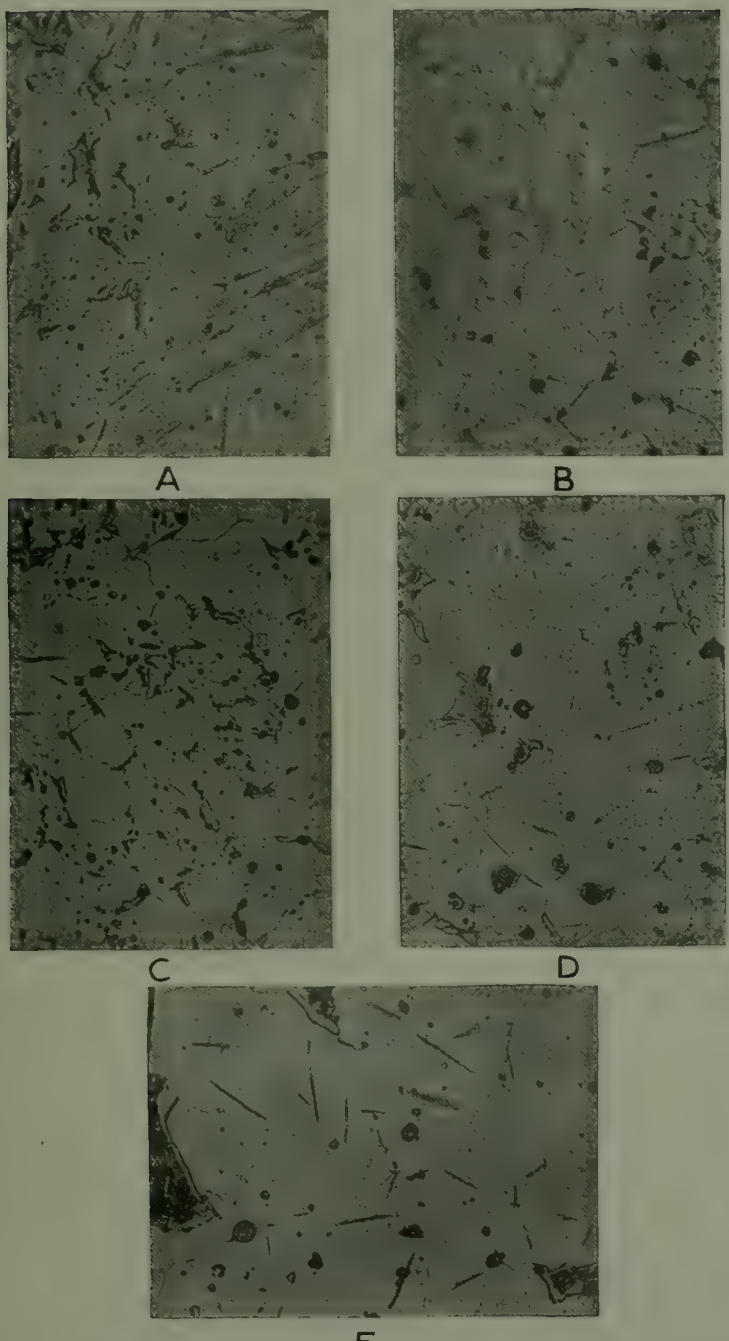


FIG. 21.—*Microstructure of arc-fused steel, Type A, showing the effect of heat treatment.* × 500

The specimen A-2 (Table 6) was quenched in water from approximately 1000° C (considerably above Ac_3 temperature, Fig. 27), and reheated as shown below. *A*, arc-fused metal as deposited; *B*, same as *A* after quenching and subsequently reheating to 650° C; no plates have formed during this treatment; *C*, a quenched specimen was reheated to 700° C; a few plates have appeared; *D*, a quenched specimen was reheated to 800° C; *E*, a quenched specimen was reheated to 925° C. Etching reagent, 2 per cent alcoholic solution of nitric acid

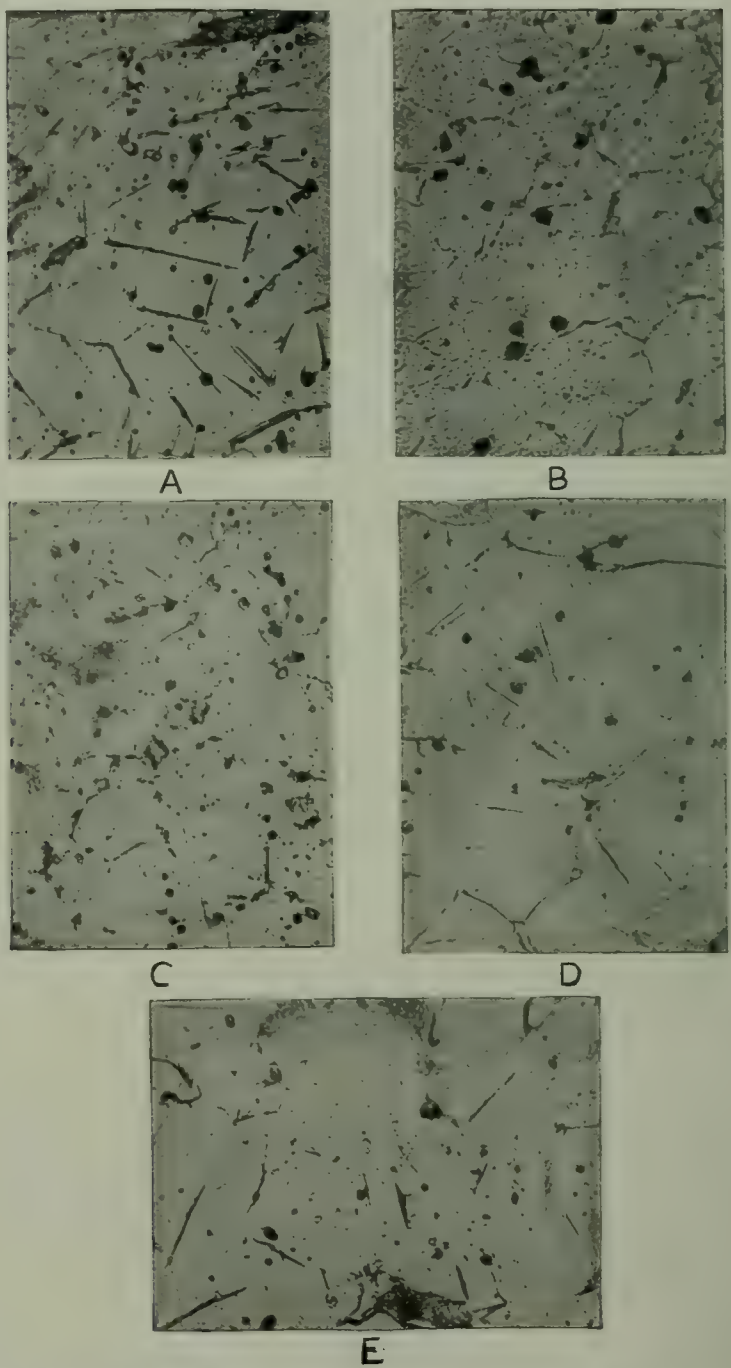


FIG. 22.—*Microstructure of arc-fused steel, type A, showing the effect of heat treatment.* × 500

The specimen AD–10 (Table 6) was quenched in water from approximately 1000° (C considerably above Ac_3 temperature, Fig. 27) and reheated as shown below. *A*, arc-fused metal as deposited; *B*, same as *A* after quenching and subsequently reheating to 650° C; no "plates" have been formed by this treatment; *C*, a quenched specimen was reheated to 700° C; a few plates have appeared; *D*, a quenched specimen was reheated to 800° C; *E*, a quenched specimen was reheated to 925° C. Etching reagent, 2 per cent alcoholic solution of nitric acid

and in the material heated to 925° C they are as large and as numerous as in any of the arc-fused specimens. The heating also develops the islands of pearlite, which are not always to be distinguished very clearly in the simple fused metal. The work of Ruder [**28**], referred to above, shows that nitrogenized iron which has been quenched and so rendered free from the nitride plates behaves in a similar manner upon heating to temperatures varying from 700 to 950° C; the plates reappear after a heating for 15 minutes at 700° C (or above), followed by a slow cooling. The similarity in behavior of the two is a further line of evidence that the arc-fused metal contains more or less nitrogenized iron throughout its mass.

The persistence of the nitride plates was also studied in specimens heated at 1000° C in vacuo for periods of six and of four and one-half hours. A set of specimens (one each of test bars A–D2, A–3, AD–6, A–10, B–2, B–4, B–5, and BD–5) was packed in a Usalite crucible, and covered with alundum "sand"; this crucible was surrounded by a protecting alundum tube and the whole heated in an Arsem furnace. A vacuum equivalent to 0.2 mm mercury was maintained for the greater part of the six-hour heating period; for the remainder of the time the vacuum was equivalent to 1.0 to 0.2 mm mercury. The specimens were allowed to cool in the furnace. Ruder [**24**] has stated that one hour's heating in vacuo at 1000° C was sufficient to cause a marked diminution in the number of plates in both arc-weld material and nitrogenized iron, and that at 1200° C they disappeared entirely.

The results obtained are shown in Fig. 25. In contradistinction to Ruder's work, the plates are more conspicuous and larger than before, and the oxide specks are larger and fewer in number. It would appear that the conditions of the experiment are favorable for a migration of the oxide through an appreciable distance and for a coalescing into larger masses. The oxide is eliminated entirely in a surface layer averaging approximately 0.15 mm in depth. Only in projections (right-angled corners, sections of threads of the tension bar, etc.) was there any removal of the nitride plates by the action of the continued heating in vacuo. This is shown in Fig. 26*c*, which illustrates the removal of the oxide inclusions also. No evidence was found that the small amount of carbon present in the arc-fused metal is eliminated, particularly very far beneath the surface.

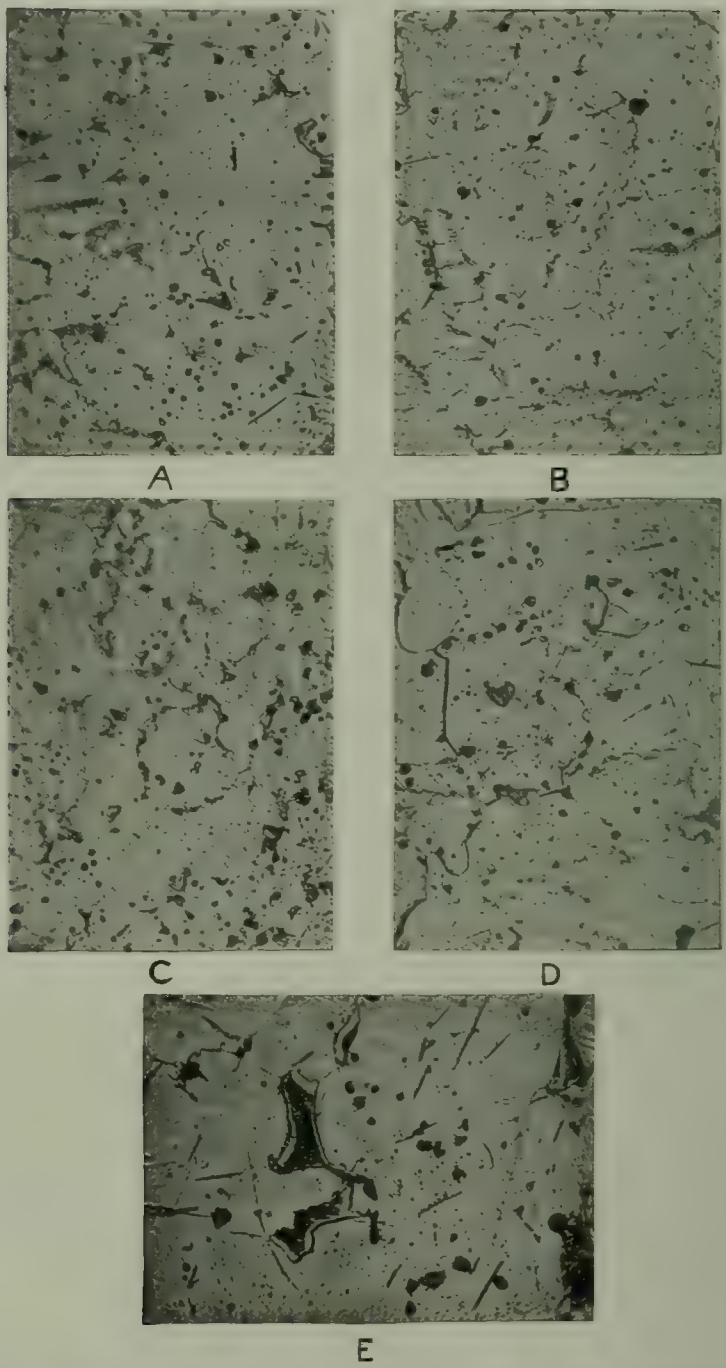


FIG. 23.—*Microstructure of arc-fused steel, type B, showing the effect of heat treatment.* × 500

The specimen B–2 (Table 6) was quenched in water from approximately 1000° C and reheated as shown below. *A*, arc-fused metal as deposited; *B*, a quenched specimen was reheated to 650° C; no plates have appeared after this treatment; *C*, a quenched specimen was reheated to 700° C; a few plates have appeared; *D*, a quenched specimen was reheated to 800° C; *E*, a quenched specimen was reheated to 925° C. Etching reagent, 2 per cent alcoholic solution of nitric acid

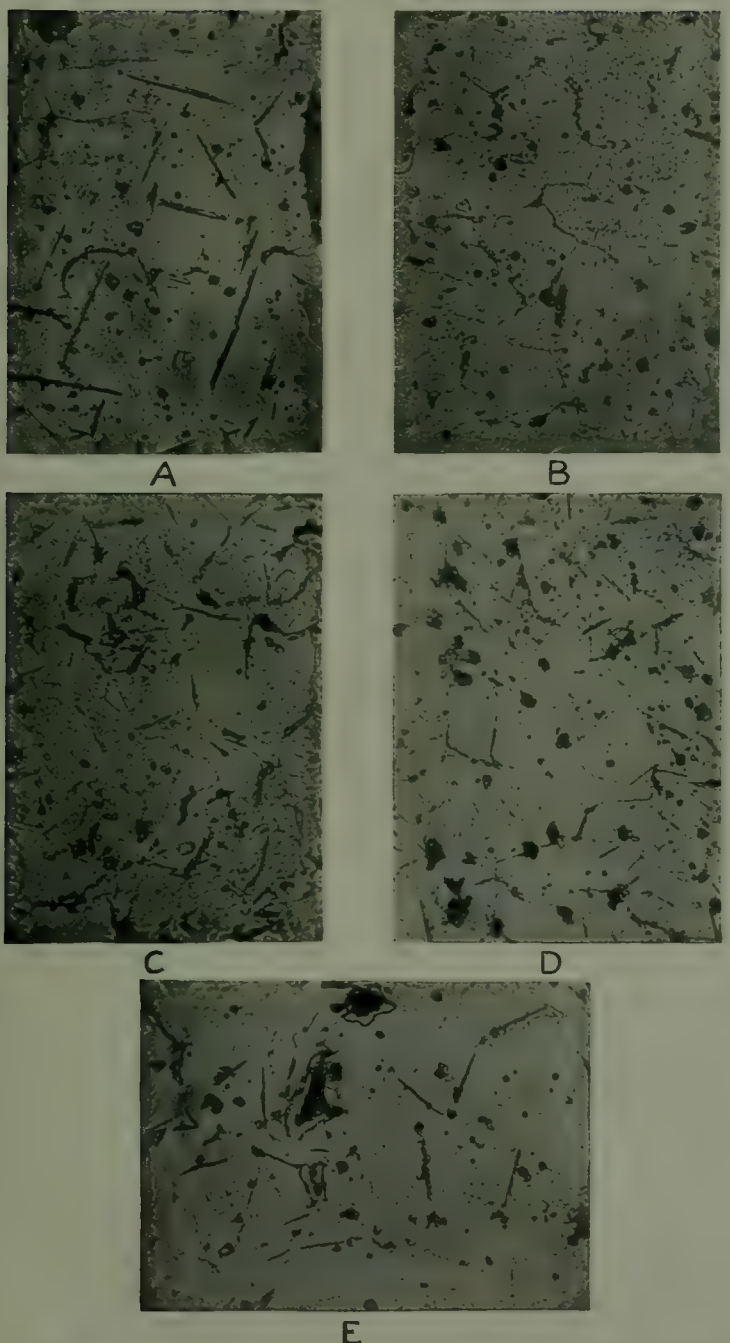


FIG. 24.—*Microstructure of arc-fused steel, type B, showing the effect of heat treatment.* × 500

The specimen B–9 (Table 6) was quenched in water from approximately 1000° C and reheated as shown below. *A*, arc-fused metal as deposited; *B*, a quenched specimen was reheated to 650° C; no plates were formed by this treatment; *C*, a quenched specimen was reheated to 700° C; a few plates have appeared; *D*, a quenched specimen was reheated to 925° C. Etching reagent, 2 per cent alcoholic solution of nitric acid

Fig. 12*c* illustrates an interesting exception to the rule that the nitride plates are flat. In the metallic globule inclusion shown the plates have a very pronounced curve. The general appearance suggests that the "metallic globules" solidified under a condition of constraint, and that this condition still persists even after the six hours' heating at 1000° C which the specimen received.

Several of the specimens which were heated in vacuo were analyzed for nitrogen. The results are given in the table below.

TABLE 9.—Change in Nitrogen Content of Arc-Fused Metal upon Heating[a]

Specimen	Weight of sample	Hours of heating in vacuo at 1000° C	Average nitrogen content		Loss
			Before heating	After heating	
	Grams		Per cent	Per cent	Per cent
A-3	1.39	6	0.127	0.062	51
B-4	6.0	6	.124	.078	37
BD-5	1.62	6	.140	.059	57
B-5	1.16	6	.121	.054	55
AD-2	1.28	10½	.149	.043	71
B-2	1.76	10½	.152	.050	67
AD-6	2.23	10½	.134	.061	54

[a] The method used for the determination of the nitrogen gives only the *nitride* nitrogen; hence a possible explanation for the change in the nitrogen content is that it has been converted into another form than nitride and may not have been eliminated as the results apparently indicate.

The fact that the specimens lose nitrogen upon heating, although the amount remaining is still many times the nitrogen content of the metal before fusion, coupled with the fact that the "nitride plates" are larger and more conspicuous after heating than before, suggests very strongly that these plates are not simple nitride of iron. In some of the specimens heated in vacuo at 1000° C for the longer period many of the crystals contain the smaller or secondary lines (Fig. 26*a*), previously referred to (p. 33). This would indicate that these secondary lines represent a transition stage. In some of the crystals (Fig. 26*b*) the lines have assumed an appearance which suggests that they are a crystalline phenomenon somewhat similar to Neumann bands.

4. THERMAL ANALYSIS OF ARC-FUSED STEEL[7]

In order to throw further light on the nature of the plates (nitride) found in the metal after fusion in the arc, the thermal characteristics of the electrode material before and after fusion as revealed by heating and cooling curves were determined.

[7] Credit is due to Howard Scott, assistant physicist, for the work described in this section.

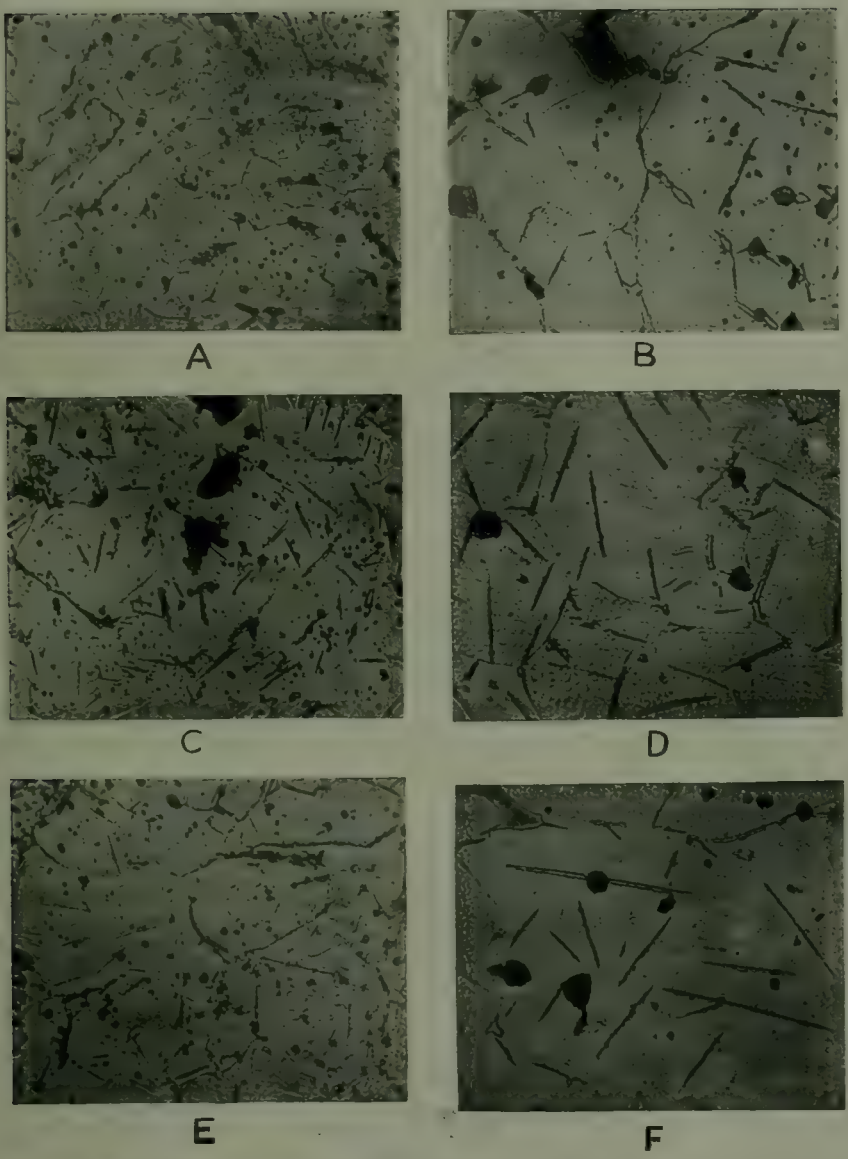


FIG. 25.—*Microstructure of arc-fused iron showing the effect of heating in vacuo at a high temperature.* × 500

The specimens were heated continuously for 6 hours at 1000° C in vacuo. *A*, specimen AD–2, initial structure; *B*, same as *A*, after heating; *C*, specimen B–4, initial structure; *D*, same as *C* after heating; *E*, specimen A–10, initial structure; *F*, same as *E*, after heating. *B*, *D*, and *F* all show some cementite at grain boundaries which resulted from the "divorcing" of pearlite. Many of the plates appear to have been influenced in their position by an oxide globule. Etching reagent, 2 per cent alcoholic solution of nitric acid

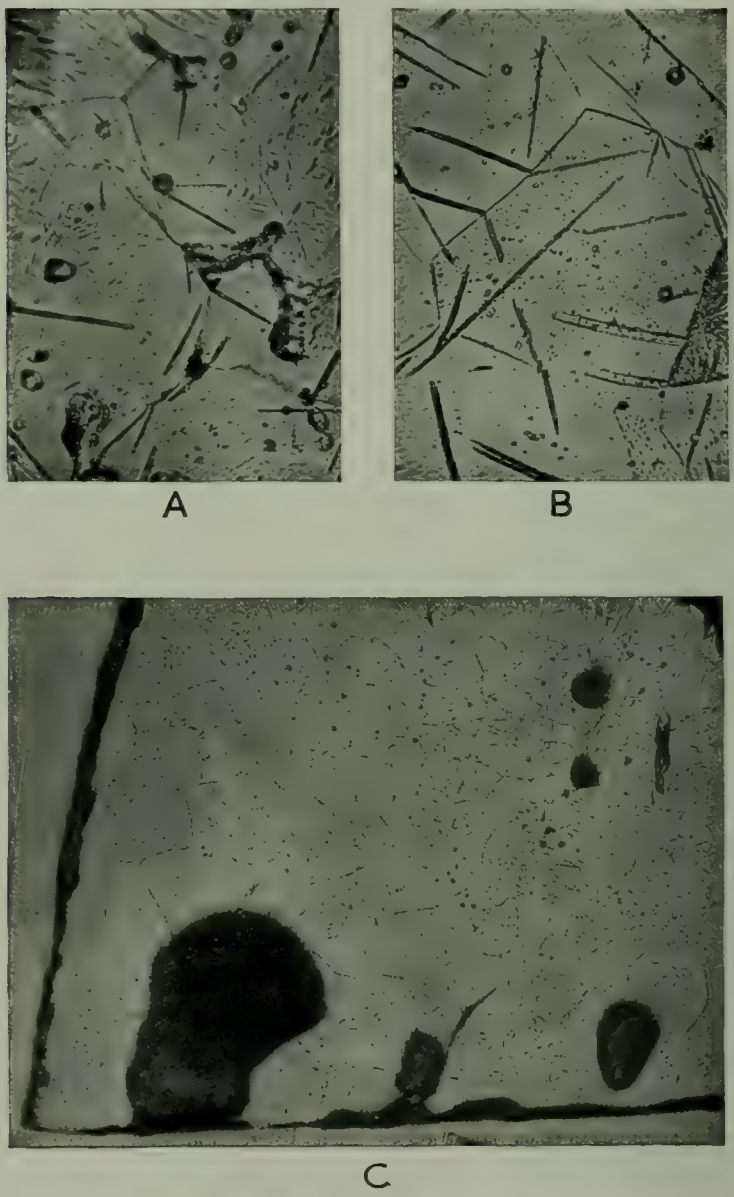


FIG. 26.—*Microstructure of arc-fused steel showing the effect of prolonged heating at high temperature*

A, specimen AD–2 (Table 6) was heated in vacuo for 10½ hours at 1000° C in stages of 6 and 4½ hours; two generations of "plates" have appeared after this treatment, × 500; *B*, specimen AD–6 (Table 6) heated as in *A* above; the plates which have persisted are relatively large and resemble Neumann bands in appearance, × 500; *C*, specimen AD–10 (Table 6) heated 6 hours in vacuo at 1000° C; the micrograph represents a corner of the specimen; the oxide and the "nitride plates" have been removed at the exposed corner, × 100

Samples of a three-sixteenths-inch electrode of type A and of the specimen, A–5, which resulted from the fusion were used as material (composition in Tables 3 and 6).

In Fig. 27 are given the curves obtained which show the characteristic behavior of the arc-fused metal upon heating. The commonly used inverse-rate method was employed in plotting the data; the details of manipulation and the precautions necessary for the thermal analysis have already been described.[8] In Table 10 are summarized the data shown graphically in Fig. 27.

TABLE 10.—The Thermal Characteristics of Arc-Fused Iron

Rate of heating and cooling	Ac_3 maximum	Ac_3				Time above A_3	Ar_3			Ar_3 maximum
		Beginning	Maximum	End	Maximum temperature		Beginning	Maximum	End	
°C sec.	°C	°C	°C	°C	°C	Minutes	°C	°C	°C	°C
UNFUSED ELECTRODE										
[a] 0.16 / [a] .15	768	892	910	918	960	.....	896	893	879	766
.15	765	897	911	916	960	.....	895	891	879	766
ARC-FUSED METAL [b]										
0.14	764	.....	847	874	960	28	847	838	820	764
.13	764	.....	849	876	935	42	847	836	822	764
.13	764	.....	844	870	910	29	847	837	821	765
.13	766	.....	850	874	1035	256	848	835	816	764

[a] Heated at rate of 0.16° C per second, cooled at 0.15° C per second; for other specimens the rate of cooling equaled the rate of heating.

[b] The same specimen was heated four times in succession, as shown.

The principal change to be noted which has resulted from the arc fusion of the iron is in the A_3 transformation. This is now very similar to the corresponding change observed in a very mild steel (for example, approximately 0.15 per cent carbon). That the difference in the A_3 transformation of the arc-fused metal as compared with that of the original electrode is not due to an increase in the carbon content is evident from the lack of the sharp inflection of the A_1 transformation ("pearlite point"), which would, of necessity, be found in a low-carbon steel. No evidence of the A_1 change was observed for the arc-fused iron within the range of temperature, 150 to 950° C. The change in the character of the A_3 transformation is without doubt to be attributed to the influence of the increased nitrogen content of the iron.

[8] H. Scott and J. R. Freeman, B. S. Sci. Papers, No. 348.

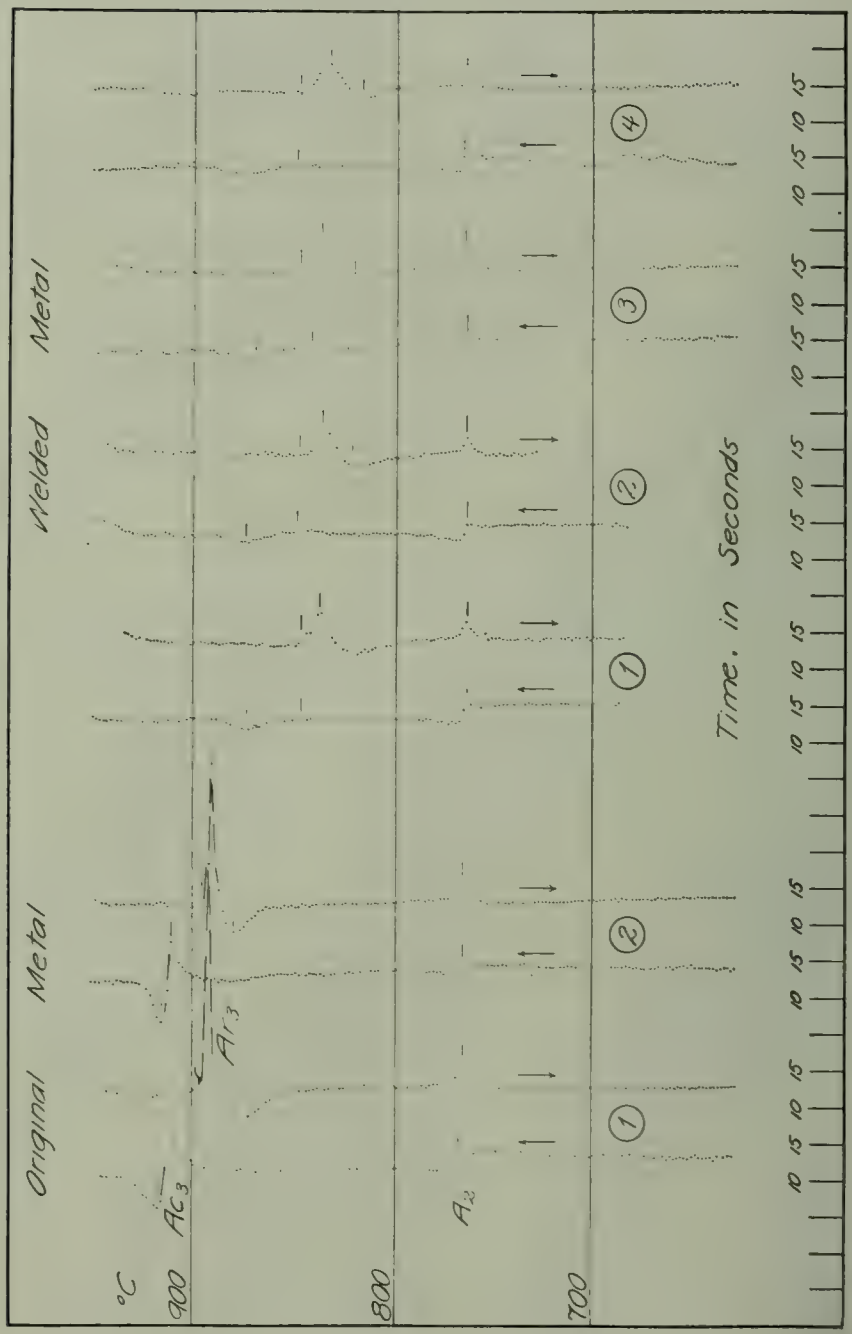
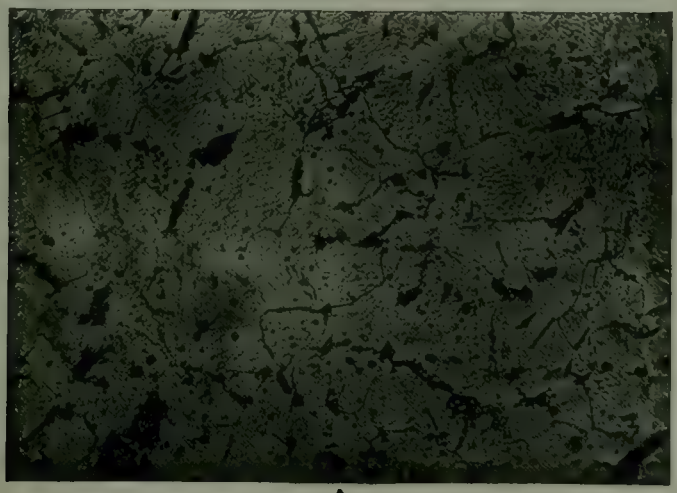


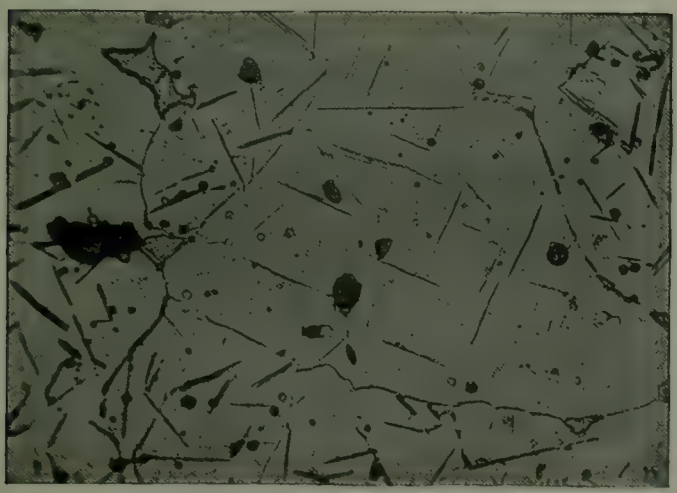
FIG. 27.—*Thermal characteristics of arc-fused steel*

The direction of arrow indicates whether the curve is a heating or a cooling curve. The numerals indicate the number of "runs" taken on the same specimen

The specimen was maintained above the temperature of the A_3 transformation for a total period (four heatings) of six hours, the



A



B

FIG. 28.—*Microstructure of the arc-fused metal used in thermal analysis.* × 500

The specimen A–5 was heated four times as shown in Fig. 27. The total length of time above the temperature of the Ac_3 transformation was 6 hours. *A*, initial structure as fused; *B*, structure after heating. Etching reagent, 2 per cent alcoholic solution of nitric acid

maximum temperature being 1035° C. The transformation apparently is unaffected by the long-continued heating, thus confirming the results described in the preceding section. In

Fig. 28 are given micrographs to show the change of structure occurring in the specimen.

In discussing the properties of steel nitrogenized by melting it in nitrogen under pressure, Andrew [**38**] states that it was found possible to extract almost entirely the small quantities of nitrogen by heating a specimen at 1000° C in vacuo for periods of one to six hours. The metal used contained 0.16 per cent carbon and 0.3 per cent nitrogen. Thermal curves are given to show that there are no critical transformations in the material; the nitrogen suppresses them. They gradually reappear, however, as the nitrogen is removed by heating the material in vacuo at 1000° C. Several days' heating was required, however, to obtain an entirely degasified product, the carbon also being removed. A further statement is made that a steel of 0.6 per cent carbon content containing 0.25 per cent nitrogen can be brought back to the normal state of a pure steel only by several weeks' heating in vacuo.

The results of the thermal analysis add considerable confirmatory evidence to support the view that the plates existing in the arc-fused metal are due to the nitrogen rather than to carbon.

VIII. DISCUSSION OF RESULTS

In any consideration of electric-arc welding it should be constantly borne in mind that the weld metal is simply metal which has been melted and has then solidified in situ. The weld is essentially a casting, though the conditions for its production are very different from those ordinarily employed in the making of steel castings. The metal loses many of the properties it possesses when in the wrought form, and hence it is not to be expected that a fusion weld made by any process whatever will have all the properties that metal of the same composition would have when in the forged or rolled condition. A knowledge of the characteristic properties of the arc-fused iron is then of fundamental importance in the study of the electric-arc weld.

The peculiar conditions under which the fusion took place also render the metal of the weld quite different from similar metal melted and cast in the usual manner. It is seemingly impossible to fuse the metal without serious imperfections. The mechanical properties of the metal are dependent, therefore, to an astonishing degree upon the skill, care, and patience of the welding operator. The very low ductility shown by specimens when stressed in tension is the most striking feature observed in the mechanical

properties of the material as revealed by the tension test. As explained above, the measured elongation of the tension specimen does not truly indicate a property of the metal. Due to the unsoundness, already described in the discussion of the structure, the true properties of the metal are not revealed by the tension test to any extent. The test measures, largely for each particular specimen, the adhesion between the successively added layers, which value varies considerably in different specimens on account of the unsoundness caused by imperfect fusion, oxide, and other inclusions, tiny inclosed cavities, and similar undesirable features. The elongation measured for any particular specimen is due largely, if not entirely, to the increase of length due to the combined effect of the numerous tiny imperfections which exist throughout the sample.

That the metal is inherently ductile, however, is shown by the behavior upon bending as recorded in the microstructure of the bent specimen. The formation of slip bands within the ferrite grains to the extent which was observed is evidence of a high degree of ductility. It appears, however, that the grosser imperfections are sufficient to prevent any accurate measurement of the real mechanical properties of the metal from being made. The conclusion appears to be warranted, therefore, that the changes of composition which the fusion entails, together with the unusual features of microstructure which accompany the composition change, are of minor importance in determining the strength, durability, and other properties of the arc weld.

The view that the characteristic features observed in the structure of the arc-fused iron are due to the increased nitrogen content is supported by several different lines of evidence. These include the likeness of the structure of the material to that of pure iron which has been "nitrogenized," the similarity in the behavior of both arc-fused and nitrogenized iron upon heating, the evidence shown by thermal analysis of the arc-fused metal, together with the fact that, as shown by chemical analysis, the nitrogen content increases during fusion while the other elements, aside from oxygen, decrease in amount. The characteristic form in which oxide occurs in iron, together with its behavior upon heating, renders very improbable the assumption that the oxide is responsible for the plates observed in the material.

In arc-fusion welds in general, the mass of weld metal is in intimate contact with the parts which are being welded, so that it is claimed by many that because of the diffusion and inter-

mingling of the metal under repair with that of the weld the properties of the latter are considerably improved. The comparison shown in the table below somewhat supports this claim. The nearest comparison found available with this Bureau's specimens are some of those of the welds designated as the "Wirt-Jones" series reported by H. M. Hobart [1]. These welds were of the 45° double-V type made in one-half-inch ship plate; the specimens for test were of uniform cross section 1 by one-half inch, the projecting metal at the joint having been planed off even with the surface of the plates and the test bars were so taken that the weld extended transversely across the specimen near the center of its length. The electrodes used were similar to those designated as type B in this Bureau's investigation.

TABLE 11.—Comparison of Mechanical Properties of Welds with Results of Tests of Arc-Fused Metal Prepared under Similar Conditions

Bureau of Standards				Wirt-Jones			
Diameters of electrodes	Direct current	Ultimate tensile strength	Elongation in 2 inches	Diameter of electrode[a]	Direct current	Ultimate tensile strength	Elongation in 2 inches
	Amp.	Lbs./in.²	Per cent		Amp.	Lbs./in.²	Per cent
1/8 inch [a]	110	52 650	7.5	1/8 inch	110	45 800	8.0
1/8 inch [b]	110	49 050	9.0	1/8 inch	115	58 200	14.0
1/8 inch [b]	110	44 400	6.5	1/8 inch	115	59 400	12.5
				1/8 inch	120	53 700	7.0
				1/8 inch	120	57 600	8.5
Average		48 700	7.7	Average		54 940	10.2
5/32 inch [a]	145	46 450	5.0	11/64 inch	150	60 900	8.0
5/32 inch [b]	145	48 130	8.0	5/32 inch	155	62 600	11.5
5/32 inch [b]	145	41 750					
Average		45 440	6.3	Average		61 750	9.8
5/32 inch [a]	185	49 600	7.5	11/64 inch	175	59 800	9.0
5/32 inch [b]	185	49 086	12.5				
5/32 inch [b]	185	47 100	11.0				
Average		48 595	10.3				

[a] Electrodes were used in bare condition. [b] Electrodes were coated as previously described.

Since the specimens used in work described in the foregoing sections were prepared in a manner quite different from the usual practice of arc welding, no definite recommendations applicable to the latter can be made. It appears, however, from the results obtained that the two types of electrodes used—that is, "pure" iron and low-carbon steel—should give very similar results in practical welding. This is due to the changes which

occur during the melting, so that the resulting fusions are essentially of the same composition. The use of a slight coating on the electrodes does not appear to be of any material advantage so far as the properties of the resulting fused metal are concerned. Since the program of work as carried out did not include the use of any of the covered electrodes which are highly recommended by many for use in arc welding, particularly for "overhead work," no data are available as to the effect of such coatings upon the properties of the metal resulting from fusion. Although all of the specimens used in the examinations were made by the use of direct current, it appears from the results obtained with a considerable number of welds representing the use of both kinds of current, submitted for the preliminary examinations, that the properties of the fused metal are independent of the kind of current and are influenced primarily by the heat of fusion. Any difference in results obtained by welding with alternating current as compared with those obtained with direct current apparently depends upon the relative ease of manipulation during welding rather than upon any intrinsic effect of the current upon properties of the metal.

IX. SUMMARY

1. A fusion weld is fundamentally different from all other types in that the metal of the weld is essentially a casting. The arc-fusion weld has characteristics quite different from other fusion welds. A preliminary study of a considerable number of specimens welded under different conditions confirmed this general opinion concerning arc welds. A knowledge of the properties of the arc-fused metal is fundamental in any study of electric-arc welding.

2. Blocks of arc-fused metal of size large enough to permit a tension specimen (0.505 inch diameter, 2-inch gage length) to be taken from each were made under different conditions of fusion. Two types of electrodes, a "pure" iron and a mild steel, were used in the bare condition and also after receiving a slight coating. With these were included a set of similar specimens prepared outside of this Bureau by expert welding operators.

3. During the fusion the composition of the metal of both types of electrodes is changed considerably by the burning-out of the carbon and other elements, and the two become very much alike in composition. A very considerable increase in the nitrogen content occurs at the same time.

4. The mechanical properties of the arc-fused metal as measured by the tension test are essentially those of an inferior casting. The most striking feature is the low ductility of the metal.

5. All of the specimens showed evidence of unsoundness in their structure, tiny inclosed cavities, oxide inclusions, lack of intimate union, etc. These features of unsoundness are, seemingly, a necessary consequence of the method of fusion as now practiced. They determine almost entirely the mechanical properties of the arc-fused metal. The observed elongation of the specimen under tension is due to the combined action of the numerous unsound spots rather than to the ductility of the metal. That the metal is inherently ductile, however, is shown by the changes in the microstructure produced by cold bending. By taking extreme precautions during the fusion, a great deal of the unsoundness may be avoided and the mechanical properties of the metal may be considerably improved. However, the specimens as described are more representative of actual present practice in welding.

6. A very characteristic feature of the microstructure of the arc-fused metal is the presence of numerous plates within the ferrite crystals, arranged usually along crystallographic planes. These persist in the metal after prolonged heating; for example, ten hours at 1000° C in vacuo does not remove them. The various lines of evidence available indicate that they are related to the nitrogen content of the metal, which is increased during the fusion.

7. Upon heating the metal above the temperature of the Ac_3 transformation, the nitride enters into solid solution in the ferrite and is retained so by quenching. Upon heating this quenched metal, the plates reappear and begin to show at a temperature of approximately 700° C. The effect of nitrogen upon the Ar_3 transformation is very similar to that of an equal amount of carbon. However, there is no evidence of a transformation corresponding to the A_1 or the pearlite change due to the nitrogen in the steel.

8. The microscopic examination indicates that there is but little, if any, relation between these nitride plates and the path of rupture produced by tension. The effect of grosser imperfections of the metal is so much greater than any possible effect of the nitride plates in determining the mechanical properties that the conclusion appears to be warranted that this feature of the structure is a matter of relatively minor importance in ordinary arc welds.

9. Judged from the results obtained, neither type of electrode appears to have a marked advantage over the other. The use of a slight protective coating on the electrodes does not appear to affect materially in any way the mechanical properties of the arc-fused metal.

10. The specimens were prepared in a manner quite different from that used ordinarily in electric-arc welding, and the results do not justify any specific recommendations concerning methods of practice in welding.

Credit is due to H. L. Whittemore and R. W. Woodward for aid in the necessary mechanical testing, and to F. H. Tucker for the general chemical analyses made.

WASHINGTON, June 7, 1920.

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DEPARTMENT OF COMMERCE

TECHNOLOGIC PAPERS

OF THE

BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 180

CAUSES AND PREVENTION OF THE FORMATION OF NONCONDENSIBLE GASES IN AMMONIA ABSORPTION REFRIGERATION MACHINES

BY

E. C. McKELVY, Chemist

AARON ISAACS, Associate Chemist

Bureau of Standards

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CAUSES AND PREVENTION OF THE FORMATION OF NONCONDENSIBLE GASES IN AMMONIA ABSORPTION REFRIGERATION MACHINES

By E. C. McKelvy and Aaron Isaacs [1]

CONTENTS

	Page.
1. Introduction	3
2. Experiments with distilled water	5
3. Experiments with aqua ammonia	6
4. Determination of carbon dioxide	7
5. Methods of preventing gas formation	8
6. Summary	10

1. INTRODUCTION

One of the great difficulties encountered in the operation of an ammonia absorption refrigeration machine is the formation of foul or noncondensing gases. The presence of these gases is usually indicated by the increase in the operating pressure of the ammonia plant. If a sample of liquor from the generator of such a plant be taken, it will always be colored a deep brown and will deposit a sediment upon standing, thereby showing that corrosion has accompanied the gas formation within the generator. However, the corrosion of the interior of the generator is of secondary importance. The foul gas causes the most trouble, since the plant must be purged of this gas just as often as it forms, with resulting loss of ammonia. The presence of foul gas makes it necessary to operate the plant at higher temperatures and pressures, thereby causing greater leakage, more corrosion, more danger from possible explosions, and increased wear on the plant. The noncondensing nature of the gas causes decreased efficiency of the compressor and consequently decreased efficiency of operation of the refrigeration plant as a whole.

In order to study the composition of the gases formed, samples from a number of plants were collected and analyzed. The per-

[1] This investigation was completed prior to the death in November, 1919, of the senior author, but the manuscript was prepared subsequently by the junior author.

13229°—20

centage composition of the different samples varied considerably, but in every case the gas consisted of nitrogen, hydrogen, and oxygen. In some cases the hydrogen content was as high as 98 per cent, while in others it was as low as 1 or 2 per cent. Where the hydrogen content was very small in amount the nitrogen content was very large, and vice versa. The oxygen was usually only a minor constituent and was never present in any appreciable quantity unless the nitrogen content was very high. It was soon recognized that the nitrogen and hydrogen must come from different sources and could not come from the decomposition of the ammonia, even though in many cases the gases were present in approximately the proportions to form ammonia. Ammonia will not decompose into its constituents under the conditions of operation of an absorption plant. However, there was a possibility of the formation of nitrides or amines. Some of the sludge from the absorption plant at the Bureau of Standards, which was giving much trouble at that time, was tested for these by dissolving in acid, making the solution alkaline and distilling into standard acid. Under this treatment ammonia would be produced if any amines or nitrides were present. There was, however, no evidence of the presence of either of these compounds. This fact pointed strongly toward the conclusion that in those plants where nitrogen, or a mixture of nitrogen and oxygen, was found in the gas, these impurities were caused by air that had leaked into the system. The action of air within such a system, therefore, was one of the points that were studied. The presence of hydrogen had to be explained in another manner, and its presence in such large quantities as were found in some plants could not be accounted for on the basis of the interaction of either pure water or pure ammonia with the iron. Therefore the effect of possible impurities in ammonia upon its corrosive action was studied in order to find out definitely, if possible, if these might be the cause of gas formation and, if so, to devise a means of overcoming the difficulty.

The experimental work consisted in trying to reproduce plant conditions in the laboratory in as simple a manner as possible and in such a way as to control at will the various factors involved. All the experiments were carried out in a bomb which consisted of a piece of 4-inch steel pipe closed at each end by a blind flange with a female recess, and clamped together by means of four bolts (Fig. 1). The bomb was provided with an outlet for aqua ammonia, consisting of a steel tube 8 inches long passing through

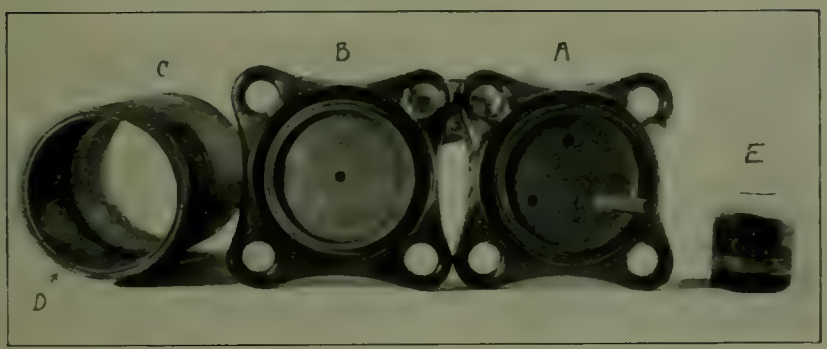

FIG. 1.—*Parts of experimental bomb used in corrosion tests*

A, top flange showing dark coating of oxide formed where gas was in contact with iron; *B*, bottom flange showing bright metal where corrosion took place under the liquid; *C*, pipe clamped between flanges showing corrosion at D where metal was under liquid; *E*, sludge formed in bomb when air was introduced into bomb containing aqua ammonia and the charge was heated.

the top flange and reaching to the bottom of the bomb. Another tube, which passed through the top flange, was used for taking gas samples. A third outlet was connected to a pressure gage. The bomb, after charging, was heated by means of an electrically heated oil bath, the temperature of which could be kept constant within a few degrees. It would have been more satisfactory in reproducing plant operating conditions, especially where air was intentionally added to the bomb, if the liquid could have been stirred or the bomb rotated; but there were so many experimental difficulties connected with doing this that it was not attempted.

The criterion of the behavior of aqua ammonia and iron toward each other was the formation of noncondensing gas, which collected mostly in the gas phase. Therefore there were in this phase ammonia, water vapor, and any noncondensing gases, such as hydrogen, oxygen, and nitrogen, that might be formed. The noncondensing gases were collected by passing the gas through a capillary tube into a gas burette inverted over sulphuric acid. It was found generally that in the gaseous phase there is practically no action between the various components and the iron. The water, or the oxygen, if air be present, in the gaseous phase reacts with the iron and forms a protective coating of magnetic oxide of iron that seems to remain unaltered until some of the liquid phase touches it; then, if this liquid is of suitable composition, considerable corrosion may take place.

2. EXPERIMENTS WITH DISTILLED WATER

Distilled water, previously heated to remove all dissolved gases, was first charged into the bomb, which was then evacuated and heated to 130° C for a period of 5 days. At the end of the experiment only a very small amount of gas had been formed, as was indicated by an increase of only 4 mm in pressure above the original pressure when the bomb was connected to a mercury gage. When the liquid was withdrawn from the bomb, it was found to be only faintly colored, and there were no signs of corrosion on the interior of the bomb. On heating with pure water to 170° C for a period of 8 days, 80 cc of gas, containing 93 per cent of hydrogen, was collected. The desirability of operating at low temperatures is indicated by the fact that as the temperature is raised the amount of hydrogen produced increases greatly.

The bomb was then recharged with boiled distilled water, and compressed air was forced into it until the pressure was about 50

pounds. In the course of a few days' heating the oxygen content of the gas was reduced to 10 per cent, the remainder of the gas being nitrogen. The bomb was opened and was found to be badly corroded, and the solution was colored a deep brown and contained considerable precipitate. The conclusion may be drawn that if air gets into a closed system of this kind the oxygen will gradually be removed and the final result will be an atmosphere of nitrogen.

An experiment was performed with water containing 0.1 per cent acetonitrile, the conditions of heating being the same as those when water was heated at 130° C. More gas was produced than with pure water under the same conditions, the pressure in this experiment rising 13 cm above the original pressure. The bomb was attacked, and the solution, although a pale yellow on withdrawal, became darker on standing and gradually changed to a colloidal solution, which finally deposited a yellow precipitate. In all cases where corrosion occurs in the absence of air there is found to be a small amount of ferrous iron in solution. The solution is nearly colorless when first removed, but on standing it becomes a deeper and deeper yellow, owing to the oxidation of the iron from the ferrous to the ferric state. Finally the iron precipitates out as a basic ferric compound.

3. EXPERIMENTS WITH AQUA AMMONIA

The experiments with water were then duplicated with solutions of pure ammonia and with the ordinary concentrated aqua ammonia of commerce, containing about 28 per cent of ammonia. In heating aqua ammonia alone, if it is pure, hydrogen is produced only by heating at high temperatures. In the experiments with either pure water or pure aqua ammonia, the rate of production of the hydrogen diminishes as the period of heating progresses. This is probably due to the fact that before beginning each experiment the interior of the bomb had been polished with emery, while at the end the interior of the bomb was covered with a firm, dense coating of oxide. However, in those cases where impurities such as carbonates or acetates are present, the rate of production of hydrogen shows no such decrease, and upon opening the bomb, the deposit on that part of the bomb in contact with the solution can be easily removed by wiping.

The experiment with air and water was also duplicated with aqua ammonia and air. In this case also the oxygen content

decreased, but not so rapidly, diminishing under the same conditions from 20 per cent to about 15 per cent. The interior of the bomb was also less attacked. A high-grade sample of aqua ammonia was used, and as the gas was in contact with a saturated solution of ammonia, the solubility of the oxygen in the solution was very greatly decreased. However, the decrease in oxygen content of the gas even in the absence of stirring was very marked.

From the above results it is seen that if air is permitted to enter the system, as may readily happen under certain faulty conditions of operation, there will result finally an atmosphere of nitrogen containing more or less oxygen. It was also found that such impurities as carbonates, acetates, and acetonitrile increase the corrosive power of aqua ammonia at ordinary operating temperatures to such an extent as to readily account for the hydrogen found in many plants.

The two impurities present in aqua ammonia that cause corrosion are, therefore, acetates and carbonates. If acetonitrile is present, it is at once saponified and converted into an acetate by the heated aqua. It is contended that because of its method of manufacture ammonia made from ammonium sulphate is the only ammonia that is free from acetonitrile. No carbon dioxide is present in anhydrous ammonia. However, examination of a number of samples of aqua ammonia showed that carbon dioxide was always present, and the amount may be increased by exposure to the atmosphere. In testing samples of aqua for corrosive properties it was found that from a determination of the carbon dioxide content the action of the aqua could be predicted. Where the content of carbonate is not over 0.003 per cent, the corrosive action is not very marked. And it was also noted that where corrosion once starts it will continue during the life of the charge.

4. DETERMINATION OF CARBON DIOXIDE

From the above evidence it is seen that one of the most important tests of aqua ammonia to be used in an absorption refrigeration machine is the determination of its carbon dioxide content. Since a content of carbon dioxide as low as 0.003 per cent will cause gas formation, a method that will permit the use of large samples, usually 100 g, was adopted.

The sample is weighed in a weight burette and is then slowly added to a glass-stoppered wash bottle containing an excess of sulphuric acid. A current of air free from carbon dioxide is

passed continually through the wash bottle and into a special wash bottle containing barium hydroxide solution. This latter wash bottle is described by E. R. Weaver and J. D. Edwards (J. Ind. Eng. Chem., 7, p. 534; 1915). When all the ammonia solution has been added, the solution is boiled for a few minutes and the air is allowed to pass through until all the carbon dioxide has been swept over into the wash bottle. The barium carbonate thus formed is washed free from barium hydroxide, dissolved in hydrochloric acid, and finally precipitated as barium sulphate. From the weight of barium sulphate thus obtained the percentage of carbon dioxide is easily calculated. Some of the results obtained are as follows:

TABLE 1.—Carbon Dioxide Determination

Sample	CO_2	
	Per cent	Per cent
C. P. reagent	0.0015	0.0016
Technical sample	.0026	.0026
Technical sample	.0075	.0071
Tap water	.0052	.0052
B. S. plant No. 1[a]	.066	.067
B. S. plant No. 2[a]	.087	.087
Calcium carbonate C. P.[b]	44.0	44.3

[a] 50 g sample used. [b] 0.1 g sample used.

5. METHODS OF PREVENTING GAS FORMATION

Among the substances which have been proposed for preventing the corrosion of iron in general is potassium dichromate solution. The authors therefore conducted experiments to determine whether a dichromate would be useful in ammonia systems. As these experiments were performed during the period of the war, sodium dichromate was used and was found to be very satisfactory. Solutions of aqua ammonia were made up to contain the various corrosive constitutents and in addition approximately 0.2 per cent by weight of commercial sodium dichromate. In all cases the amount of gas produced was very small, and less than that given by the best sample of ammonia solution that had been tested without the addition of dichromate. The solution after withdrawl from the bomb had a green color which did not change on standing. There was always a precipitate present which varied from a grayish green when pure samples of aqua ammonia were used to dark brown when the dichromate was added to the contents of a bomb in which corrosion had already

taken place. The addition of dichromate always stopped the gas formation, and upon opening the bomb the characteristic hard dull coat was observed. From these experiments in the laboratory it was decided to try the addition of sodium dichromate to the ammonia in a plant which was giving trouble on account of foul gas formation.

Much trouble had always been experienced in the operation of the 30-ton Carbondale absorption plant at the Bureau of Standards. Gas was being formed continually, and this caused large losses of ammonia in purging the system of foul gas, as well as losses due to leakage caused by the high-working pressure necessary because of the presence of the noncondensing gas. The ammonia charge at this plant became depleted during the winter 1916–17, and it was found necessary to partially recharge it. Analysis was made of the ammonia solution that still remained in the machine, and it was found to contain 19 per cent of ammonia and about 0.066 per cent of carbon dioxide. In recharging this system tap water, containing about 0.0052 per cent total carbon dioxide, and anhydrous ammonia were used. After recharging the system and operating for a few months, the carbon dioxide content had increased to 0.087 per cent and there was considerable corrosion and gas formation. The analyzed samples of liquor when tested in the experimental bomb were found to cause a great deal of corrosion and gas formation, but when sodium dichromate was added gas formation was stopped.

In June, 1917, an amount of sodium dichromate, equal to 0.2 per cent of the total weight of the aqua ammonia charge, was added in the form of an aqueous solution to the plant. No trouble has since been experienced with the plant from gas formation. At one time when the engineer reported that considerable gas was being formed in the plant, the gas was found upon analysis to consist of over 95 per cent nitrogen with a few per cent of hydrogen and a small amount of oxygen. It was soon found that this gas was caused by leaks around the piston rod of the ammonia pump. On replacing the worn-out rod gas troubles ceased and have not been experienced since. This plant is now giving better service than for many years past.

6. SUMMARY

1. The noncondensible gases found in ammonia absorption refrigeration machines are due to either or both of two causes, namely, (*a*) leaks of air into the system and (*b*) the corrosive action of the ammonia liquor on the metal of the plant.

2. When the foul gas is mainly nitrogen, the gas is derived from air that has leaked into the system, and leaks should therefore be sought. The oxygen in the air is very quickly used up and so will be present in only a very small percentage of its original amount. If the foul gas is hydrogen, the cause is corrosion by the ammoniacal liquor. A gas containing both nitrogen and hydrogen shows both causes to be present.

3. If a solution of sodium or potassium dichromate is added to the generator charge so that the charge in the generator will contain the salt to the extent of 0.2 per cent by weight, all foul gas formation from the corrosive action of the ammonia charge will be stopped. It is recommended that the dichromate be added in all cases, as it has been found that its presence decreases the very small amount of gas caused by even the highest grade ammonias.

4. A method is given for the quantitative estimation of carbon dioxide in ammonia.

WASHINGTON, July 19, 1920.

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TECHNOLOGIC PAPERS

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S. W. STRATTON, DIRECTOR

No. 181

COLORED WALL PLASTER

BY

WARREN E. EMLEY, Chemist
CHARLOTTE F. FAXON, Laboratory Assistant
Bureau of Standards

DECEMBER 15, 1920

PRICE, 5 CENTS

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1920

FIG. 1.— *Examples of the new type of colored wall plaster.*

COLORED WALL PLASTER

By Warren E. Emley and Charlotte F. Faxon

The natural color of a wall plaster is white, or nearly so. For many years it has been customary to accept this white color as inevitable and to dismiss the subject without further consideration.

The quality of a plaster is indicated by its natural color in a way which is strikingly analagous to the method used for classifying diamonds. The best plasters are slightly tinted with blue; pure white plasters are of excellent quality, and the poorest grades have a buff or reddish tinge. This fact has led the plasterer and the building contractor to look with favor upon a white or bluish-white plaster because these colors are usually accompanied by excellence in quality.

On utilitarian grounds, plaster has established its position in the building industry. Its fire resistance, its heat insulating, and its acoustical properties are of primary importance. The appearance of the plaster, while admittedly and rightfully of secondary importance, is receiving more and more attention from progressive architects and builders.

The white color of plaster is sometimes objectionable in that it does not fit in with the artistic scheme which the architect is trying to evolve. It is true that the plaster may be covered with kalsomine or wall paper, but it seems to be worth while to attempt to make the plaster of the desired color in the first place.

Several attempts have been made to produce colored plaster, but the method has not proven sufficiently satisfactory to warrant its general adoption. It involves the use of a mineral pigment, which is mixed with the plastering materials. A buff or red can be obtained with oxides of iron; a gray with lampblack. It has been found extremely difficult to mix the pigment with the plaster in such a way that two batches have exactly the same tint. Deep shades of color require the addition of so much pigment that the strength of the plaster is impaired. A plaster made by this method has a dead color, which is desirable when the plaster is considered as a background for other decorations, but which has little artistic value of itself.

14946°—20

Some years ago J. J. Earley developed on a commercial scale the manufacture of colored concretes by the exposed aggregate method. Many examples of the beauty of such concretes and of the flexibility of the process can now be seen in buildings, walls, and pavements of Washington. Instead of the usual crushed stone or gravel which is used as an aggregate in concrete, he uses crushed stone which is prepared from naturally colored rocks, carefully selected and mixed to produce the desired artistic effect. After the concrete has set, the surface is removed and the colored aggregate thereby exposed. At Mr. Earley's suggestion this same principle has been applied to the manufacture of a colored wall plaster.

The first step was the selection of the proper aggregate. Fortunately, this offered no difficulty. There has been on the market for many years a material known as "gypsum wood-fibered plaster." This consists of approximately 85 per cent of calcined gypsum (plaster of Paris), 14 per cent of some material such as lime or clay added to give it the necessary plasticity, and 1 per cent by weight of wood fiber. It is one of the many commercial forms of wall plaster, and its position in the trade is so well established that tentative specifications for it have been adopted by the American Society for Testing Materials.

This plaster is usually designed to be used without sand, the wood fiber being considered as an aggregate. The fiber is quite similar to the ground wood used in making newspaper, except that the staple is a little longer.

The proposed process consists briefly of dyeing the fiber the desired color with aniline dyes, mixing it with the other ingredients, applying the plaster to the wall in the usual way, and then, after the plaster has set, removing its surface in such a way as to expose the colored fiber.

The first difficulty encountered arose from the lack of literature describing the behavior of aniline dyes on wood. Most of our experimental work was devoted to this phase of the subject—the testing of a large number of dyes. These tests were conducted by exposing half of a sample of dyed wood fiber mixed with the other ingredients of a colored plaster to direct sunlight for six weeks. The other half of the same specimen of plaster was kept covered. If, at the end of six weeks, one could not distinguish between the two halves of the sample, the dye was pronounced satisfactorily fast to light. Fastness to water is not essential; in fact, it is usually desirable that the dye shall "bleed" a little.

The color runs into the plaster surrounding the fiber, thereby making the fiber appear to be larger than it is and making it possible to obtain the desired effect with the use of less fiber than would be possible if the dye were fast to water.

It was found that wood fiber acts quite similarly to jute in taking up dyes. Any one of the classes of dyes can be used—silk, wool, cotton, or direct. A dye which is fast to light on the fiber on which it is intended to be used will usually be fast to light on wood fiber. If the dye is fast to water, it can sometimes be made to bleed slightly by drying the dyed fiber, without washing it. As a general rule, a small amount of alum added to the dye bath answers the purpose of a mordant. The heterogeneous character of the wood prevents the dye from being absorbed evenly. As will be shown in the following discussion, this is an advantage rather than a detriment, so that no leveling agent is necessary.

A general method of procedure may be illustrated as follows: In an indefinite quantity of water, dissolve an amount of dye equal to 3 per cent of the weight of the fiber. Heat the solution nearly to boiling and add the fiber. Add about 2 per cent of alum (aluminum sulphate) based on the weight of the fiber. Digest hot for about one hour and let it cool. Remove the fiber and dry it (usually without washing) in a drying oven. These directions are to be taken in a very general sense. Changes in them, to suit the different dyes, will be suggested by the directions given by the dye manufacturers, by standard textbooks, and by experience.

The kinds of dyes which may be used are illustrated by the following list of a few of the many dyes we have found satisfactory: Alizarine cyanine green G extra, alizarine cyanine yellow, alizarine cyanine irisol, crocein scarlet 3 B, rhodamine B extra, sulphur blue, diamine brown, diamine rose, naphthol orange, beta naphthol yellow, flavopurpurine, alizarine tartrazine, chloramine brown. Obviously any desired color can be obtained.

It is to be understood that these colors have proven fast to light when tested by the laboratory method described above. Before attempting any commercial application, it would, of course, be wise to determine the permanency of the dyes which it is proposed to use. This information may be obtainable from the dye manufacturers, or it may be necessary to learn from experience. Attention is called to the fact that the color need not be perfectly fast to light to give satisfactory service. If a wall is evenly exposed, so that all parts of it fade at the same rate, the fading is not a serious defect. There is also the probability that the walls will be redecorated every few years to suit the tastes of new occupants of the building.

There are a great many pure chemical compounds which have definite colors in and of themselves. A massive piece of such a compound will appear in its true color, irrespective of the viewpoint of the observer. However, most of the materials with which we are familiar are not simple substances, but are mixtures of several ingredients. This is particularly true of wall plaster. If the different ingredients have different colors, as is usually the case, then the color of the mixture will depend upon the relative amounts of the ingredients, the relative sizes and positions of the particles, and the viewpoint of the observer. A plaster which appears gray on casual observation may, on close inspection, be found to consist of a number of black specks embedded in a white matrix. If the black specks are large enough to be easily visible, then the plaster is no longer gray, but is a mottled black and white. It will be noted that the term "easily visible" defines the position of the observer. The mottled effect, which is pleasing to the eye, is obtained only when the black particles are uniformly distributed; if they are collected in large aggregates, the effect is blotchy and displeasing.

While fibers of one color only can be used, better effects can be obtained by the use of two or more different colors. In this way, the predominating color can be made more agreeable and satisfying by the use of supporting colors. Spots of the principal color should be distributed uniformly (but not in regular pattern) throughout the matrix. They should be large enough so that their identity can be distinguished without effort. Each spot should be set off by surrounding it with a number of small spots of one or more secondary colors. These should be so small that they can not be distinguished except by close inspection. The effect of the whole can be improved by giving the surface a rough texture.

Of course, due care must be taken that only those colors are used together which harmonize well with each other.

Plasters colored by this method will have "life." By this it is meant that the color of the plaster is more or less dependent upon the position of the observer; the color changes as he moves. Suppose, as the simplest case, that a plaster contains fibers of two principal, but no secondary, colors. When viewed from one direction, it is probable that one color would be reflected much more than the other and would become dominant. Viewed from another angle, the reverse might be the case. Close inspection would reveal the two colors equally. At a sufficient distance, the reflected colored lights would be superimposed, giving rise to an

entirely different color, or even to gray if the original colors are complementary.

A similar live effect can be produced by a rough surface. The protuberances cast shadows on the depressions, and thus produce light and dark tints. The intensity and the apparent magnitude of these shadows depend upon the position of the observer, and seem to vary as he moves. The life of such a surface, and its pleasing effect, is dependent upon its texture.

The above statements form a brief outline of the principles underlying the manufacture of this type of colored plaster. It will be noted that the effect desired may be described as a "uniform lack of uniformity." It is desirable that the wood fiber shall not take up the dye evenly. The principal color can be made predominant either by using a large quantity of fiber or else by using a dye which will "bleed." For the secondary colors, the fiber should be quite small and the dye fast to water, or, if the dye will bleed, the fiber may be replaced by sawdust.

The desired proportions of the different colored fibers are mixed with the calcined gypsum and other ingredients to form a gypsum wood-fibered plaster of the usual composition. This is shipped dry, mixed with lime putty and water, and applied in the usual way. Up to this point the only novelty in the manufacture of a colored plaster lies in the use of a dyed wood fiber instead of the naturally colored material. Of course care must be exercised in the selection of colors which will blend harmoniously, and in the admixture of the proper proportions of primary and secondary colors.

The desired texture of the finished surface may call for a variation in the quantity of fiber used. It will be recalled that commercial wood-fibered plaster contains about 1 per cent of fiber by weight. This is equivalent to about 15 per cent by volume. About 1 part of fiber to 1 of plaster, by volume, is the maximum amount which can be used if the surface is to be troweled smooth. If, however, a rough surface is desired, the quantity of fiber may be increased; 2 parts of fiber to 1 of plaster can be used by patting it into place.

A rough texture is produced by brushing the plaster just as it sets. This removes the gypsum from the surface and exposes the colored fibers. Differences in the quantity of fiber and in the method of brushing permit the production of a great variety of textures. We have developed three types of texture which are illustrative of variations obtainable. The "smooth finish"

is obtained by the use of a plaster containing 0.3 to 1 part by volume of fiber to 1 part of plaster. The plaster is troweled to a smooth surface in the usual way. Since this surface is not brushed, and the fiber is not exposed, it is necessary to use dyes which will bleed rather profusely. Otherwise the color will not be sufficiently pronounced. Just before the plaster has set, it may be brushed with a fiber scrubbing brush, taking care that the marks made by the brush are all either horizontal or vertical. This produces an "organdie" finish. For the "tapestry" finish, we use 2 parts of fiber to 1 of plaster. This is patted into place but can not be troweled. Just before the plaster sets it is stippled with a wire brush. The resultant surface is very rough and bears some resemblance to tapestry. These types are illustrated in the colored photograph which accompanies this paper.

It was found that a slight efflorescence appeared during the drying of the plaster, which marred the brilliance of the colors. This was overcome by washing the plaster with soap and water. The soap can be applied just before the plaster is finally troweled down, or the plaster may be allowed to harden and dry before scrubbing it. A thick solution of strong laundry soap in hot water is recommended. Apparently the soap enters into chemical reaction with the lime in the plaster forming a calcium resinate or similar gelatinous compound, which effectually closes the pores of the plaster and prevents further efflorescence.

This method of scrubbing the plaster with soap and water affords a housewife a means of brightening the walls whenever necessary.

No matter how pleasing the appearance of a wall may be, it is probable that a change in the decorative scheme will sometime be desired. When this occurs, it is only necessary to remember that a colored wall plaster is essentially the same as any other wall plaster. It may be covered with another coat of plaster, either colored or white, or it may be painted or papered in the usual way.

In conclusion, it may be stated that a method has been developed for producing a colored wall plaster of any desired color or texture. Effects can be produced with this plaster which are not attainable with either paint or wall paper. A wall finished in this plaster can be washed when the colors become dull or soiled, or it can be redecorated in the same way as any other plastered wall.

WASHINGTON, August 16, 1920.

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EFFECT OF REPEATED REVERSALS OF STRESS ON DOUBLE-REINFORCED CONCRETE BEAMS

BY

W. A. SLATER, Engineer-Physicist

G. A. SMITH, Associate Engineer-Physicist

Bureau of Standards

H. P. MUELLER, Junior Structural Engineer

Concrete Ship Section

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CONTENTS

	Page
I. Introduction	4
1. General statement	4
2. Scope of tests	5
II. Testing apparatus	7
1. Testing machine	7
2. Standardization of testing machine	8
3. Measuring instruments	9
III. Test specimens and testing	10
1. Description of test beams	10
2. Materials	12
3. Making and storage of test specimens	15
4. Control specimens	16
5. Gage lines	16
6. Method of testing	16
IV. Test data and discussion	20
1. Tables and diagrams	20
2. Behavior of beams during tests	22
3. Significance of tension failures	34
4. End slip of bars	35
5. Effect of gage holes on location of point of failure	38
6. Effect of crack development on location of point of failure	39
7. Effect of repetition of bending stresses on physical properties of steel reinforcement	40
8. Chemical composition of steel reinforcement	41
9. Effect of rest periods on number of cycles that caused failure	41
10. Relation between magnitude of bending stresses and number of cycles that caused failure	43
11. Position of neutral axis	45
12. Summary	50

LIST OF TABLES

1. Schedule of beams for reversal-of-stress tests	11
2. Physical properties of reinforcing steel of beam 5A1	12
3. Physical properties of reinforcing steel of beam 5B1	13
4. Physical properties of reinforcing steel of beam 5F1	13
5. Physical properties of reinforcing steel of beam 5L1	14
6. Comparison of representative physical and chemical tests of reinforcing steel used in concrete ships and reversal-of-stress beams	14
7. Test results for control cylinders	16
8. Measured dimensions and computed properties of the test beams	20
9. Results of reversal-of-stress tests	21
10. Widths of cracks at points of failure	39
11. Comparison of properties of reinforcing steel after repeated loading with properties before repeated loading	40

LIST OF FIGURES

		Page
1.	Reversal-of-stress testing machine	6
2.	View of unit No. 1 of reversal-of-stress testing machine ..... facing..	8
3.	View showing the three units of reversal-of-stress testing machine.. facing..	8
4.	View showing beam 5C1 in unit No. 3 of testing machine with deflectometer in position ..... facing..	9
5.	Dimensions and reinforcement of test specimens	10
6.	Strainagraph record showing characteristic behavior of beams during test..	18
7.	View of cross sections through beams 5A1 and 5B1 after test..... facing..	18
8.	View of cross sections through beams 5L1 and 5F1 after test..... facing..	18
9.	Side view of beams 5A1 and 5B1 after test ..... facing..	19
10.	Sketch showing location of cracks on beam 5C1 after test	19
11.	Side view of beams 5F1 and 5L1 after test ..... facing..	20
12.	Deflection and total crack widths for beam 5A1	23
13.	Deflection, crack widths, and continuity of test for beam 5B1	23
14.	Deflection and crack widths for beam 5C1	24
15.	Deflection and crack widths for beam 5F1	25
16.	Deflection and crack widths for beam 5L1	26
17.	Unit deformations in bottom of beam 5A1	27
18.	Unit deformations in top of beam 5A1	28
19.	Unit deformations in beam 5B1	29
20.	Unit deformations in beam 5C1	30
21.	Unit deformations in bottom of beam 5F1	31
22.	Unit deformations in top of beam 5F1	32
23.	Unit deformations in beam 5L1	34
24.	End slip of reinforcing bars for beam 5L1	36
25.	Continuity of tests for beams 5A1, 5F1, and 5L1	42
26.	Ratio of rest periods to total time of test	43
27.	Relation between tensile stress and number of cycles at failure	43
28.	Relation between sum of compressive and tensile stresses and number of cycles at failure	44
29.	Strain distribution and positions of neutral axis for beam 5A1	46
30.	Strain distribution and positions of neutral axis for beam 5B1	46
31.	Strain distribution and position of neutral axis for beam 5C1	47
32.	Strain distribution and positions of neutral axis for beam 5F1	47
33.	Strain distribution and positions of neutral axis for beam 5L1	48

I. INTRODUCTION

1. GENERAL STATEMENT

In a ship at sea the change from "hogging" to "sagging" may occur 10 to 15 times per minute. This means a large number of reversals of stress during the life of a ship, and it means that both the deck and the bottom of the ship are required to withstand tensile and compressive stresses alternately. The effect of these reversals of stress has a bearing upon the use of reinforced concrete in lieu of wood and steel in the construction of ships, and the investigation of this effect is a matter of importance.

Although earlier tests[1] have been reported showing the effect of repeated loads in one direction on a concrete beam, none are known to have been made in which a concrete member has been subjected alternately to compressive and tensile stresses large enough to crack the concrete. Assuming that the action above described would sometime produce cracks in the structure of a concrete ship, the possibility was foreseen that the repeated opening and closing of the cracks might produce a grinding action on the surface of the fracture which would result ultimately in disintegration. This was one of the important features considered in the tests here reported. This investigation was undertaken by the Bureau of Standards at the request of the concrete ship section of the Emergency Fleet Corporation.

Acknowledgment is made to A. S. Merrill, engineer of the Bureau of Standards, for his editorial work upon this paper and for many valued suggestions and criticisms for its improvement.

2. SCOPE OF TESTS

The scope of this investigation was intended to bring out only the outstanding features of the behavior of reinforced concrete beams subjected to repeated reversal of stress. The original program was modified somewhat after the results of the first tests were obtained.

In the investigation only five beams were tested. One of these five beams had not failed at the time that it became necessary to discontinue the testing operations. However, since this beam had undergone a large number of repetitions of a load which produced approximately the design stresses, it furnishes information of some value.

Although the tests which have been carried out are considerably smaller in number than was planned, the impossibility of completing the program in the near future makes it desirable that the results which have been obtained be presented at the present time.

---

[1] J. L. Van Ornum, The Fatigue of Concrete, Trans. Am. Soc. C. E., 58, p. 294, 1907; W. K. Hatt, Notes on the Effect of Time Element in Loading Reinforced Concrete Beams, Proc. Am. Soc. Test. Mat., 7, p. 421, 1907; H. C. Berry, Some Tests of Concrete Beams under oft repeated Loading, Proc. Am. Soc. Test. Mat. 8, p. 454, 1908.

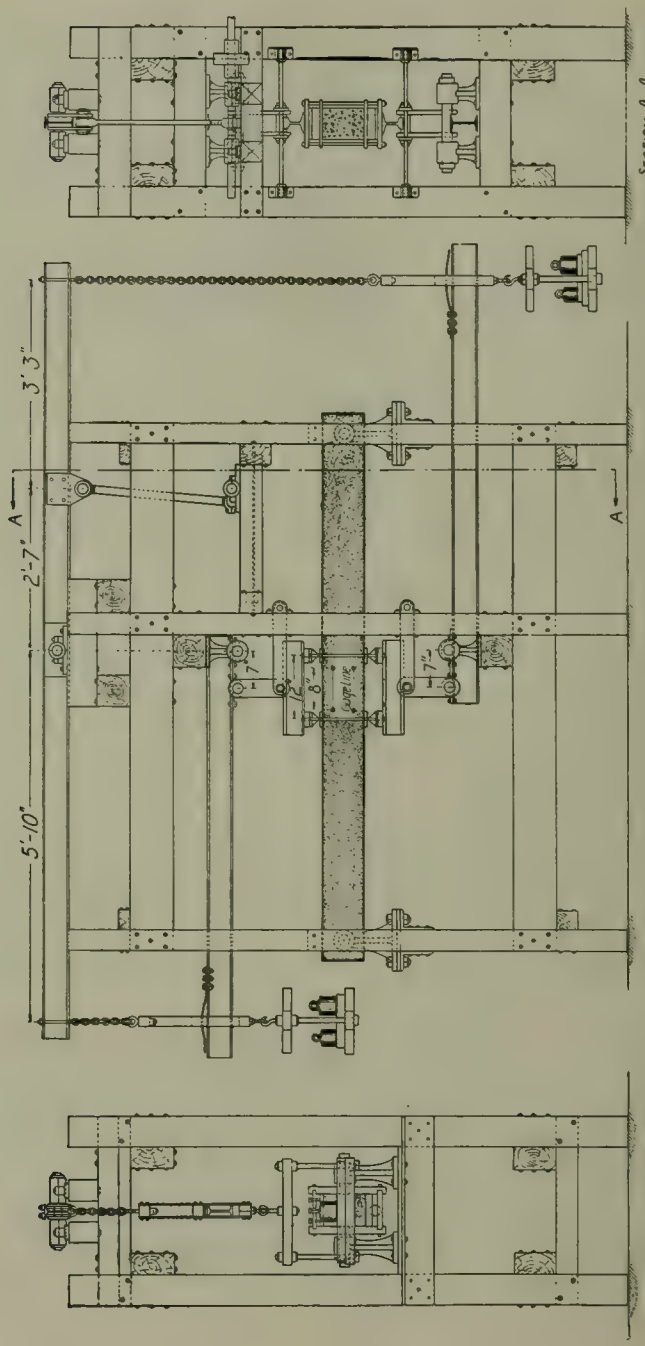

FIG. 1.—*Reversal-of-stress testing machine*

II. TESTING APPARATUS

1. TESTING MACHINE

The machine designed for these tests applied a load alternately downward and upward at a rate of approximately 17 cycles[2] per minute. Three units of the machine were built so that three beams might be under test at the same time.

The testing machine is shown in Fig. 1, and consisted essentially of a wooden frame with a system of levers so arranged and operated that a load applied at the end of one of the main levers produced a load ten times as great on the concrete test beam. One main lever applied the load in the upward direction and the other main lever applied it in the downward direction. When one load was being applied, the load in the opposite direction was being removed. Slightly before one load had been entirely removed the full load in the opposite direction had been applied.

During the test the beam was supported at sections 4 inches from each end by means of steel yokes, shown in Fig. 1. Each yoke was supported at mid height by a shaft which was free to turn as the beam deflected. Since the supporting shaft was approximately at the neutral axis, the change in the distance between the point of support, due to changing the curvature of the beam, was negligible.

The load was transmitted from each main lever through a link to a rail section, as shown in Figs. 1 and 2. By means of the rail section the load was applied to the beam at points usually 6 inches on either side of the center of the span both on the top and on the bottom of the beam, but in testing beam 5L1 the knife-edges were placed 12 inches either side of the center. The knife-edges through which the load came upon the beam were held in place by means of a yoke around the beam at each point of application of load

When tests were being conducted, the units were driven by means of a shunt-wound electric motor developing 3-horsepower at 1700 revolutions per minute. The shaft of the motor is directly connected to a worm-gear reducing unit. An individual shaft, clutch, and pulley arrangement enables each unit to be operated independently. The walking beam, which applies the load to the main levers, is driven by means of a connecting rod from a crank attached to the individual driving shaft of each unit. A

[2] The term "cycle," as used in this report, refers to one complete revolution of the crank shaft of the testing machine. Each cycle involved one application of the load downward and one application of the load upward.

control rheostat regulates the speed of the motor at different loads and is the means of obtaining the same speed whether running with one unit or all units in operation. For a part of the time that the tests were in progress the machines were run continuously day and night.

Fig. 3 shows the position of the three units of the machine relative to each other and to the driving motor. Fig. 4 is a side view of a beam in one of the units of the testing machine.

2. STANDARDIZATION OF TESTING MACHINE.

A standard 6-inch I-beam was used to rate the reversal-of-stress testing machine. The I-beam was first standardized in a 200 000-pound Olsen testing machine, using an 8-foot span and 2-point loading, 6 inches either side of mid span. Loads were applied in increments of 500 pounds, as measured on the scale beam of the Olsen machine, and the corresponding deflections were measured at mid span. Readings of deflection were also noted as the load was removed. The beam was then inverted and loaded again, and it was found that the load-deflection curve agreed closely with that for the beam loaded in the previous position. The extreme fiber stress did not exceed the elastic limit of the steel. The I-beam was then transferred to the reversal-of-stress machine and loaded with the same spacing of load points and supports as that employed in the Olsen testing machine and in the tests of the reinforced concrete beams.

From the deflection curve obtained from the test in the Olsen machine and the deflection curve from the loading in the reversal-of-stress machine, curves were drawn which gave the relation between the applied load and the load on the weight carrier. It was found that the equations of these curves varied slightly for the different units. The equations found were corrected for the weight of the beam and for certain parts of the loading apparatus and were used in determining the loads on the test specimens. The equations are as follows:

Unit 1 upward $W = 2088 + 10.03\ W_1 - \frac{4}{7} W_2 - W_3$.
Unit 1 downward $W = 2070 + 9.79\ W_1 + \frac{4}{7} W_2 + W_3$.
Unit 2 upward $W = 2078 + 9.88\ W_1 - \frac{4}{7} W_2 - W_3$.
Unit 2 downward $W = 1990 + 10.05\ W_1 + \frac{4}{7} W_2 + W_3$.
Unit 3 upward $W = 2070 + 9.83\ W_1 - \frac{4}{7} W_2 - W_3$.
Unit 3 downward $W = 2000 + 9.41\ W_1 + \frac{4}{7} W_2 + W_3$.

FIG. 2.—*View of unit No. 1 of reversal-of-stress testing machine*

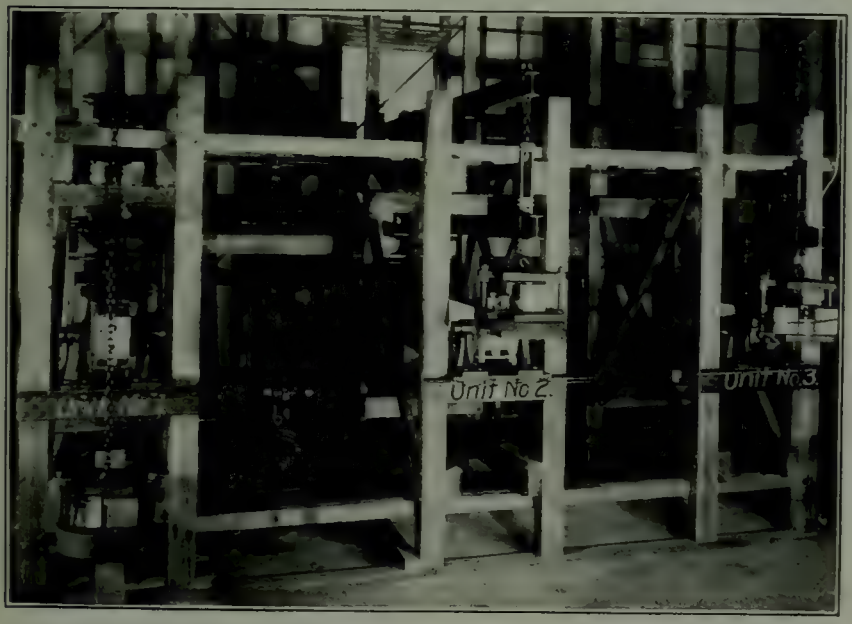

FIG. 3.—*View showing the three units of reversal-of-stress testing machine*

FIG. 4.—*View showing beam 5C1 in unit No. 3 of testing machine with deflectometer in position*

In these equations W is the load upon the test beam, W_1 is the load applied to the weight carrier, W_2 is the weight of the test specimen, and W_3 is the weight of the yokes and T-rails used for applying the load.

It will be seen that for all the units the load without weights being added to the weight carriers was about 2000 pounds. In testing beam 5C1 it was desired to apply only 670 pounds upward. This necessitated removing the weight carrier at one end of unit No. 3 and the attaching of small weights to the main lever. This may be seen at the right of the view in Fig. 4.

3. MEASURING INSTRUMENTS

A Berry strain gage, having an 8-inch gage length, was used to measure the deformations in the steel reinforcement. The multiplication ratio of this strain gage was 4.94. Readings on a gage line on an unstressed bar of structural steel were used as a basis for correcting all strain-gage readings on the test specimens.

For measuring deflections a removable deflectometer was used. It consisted of a wooden bar having fixed trammel points 8 feet apart and having an Ames-gage micrometer mounted at its center. The wooden bar was supported on the steel yokes at the ends of the test beam in the same position for consecutive observations. The difference in the deflections at the center of the span was shown by the Ames gage. Readings were made directly to the nearest 0.001 inch. To provide for the exact placing of the deflectometer, one end was held in a gage hole on one supporting collar, while the opposite end was held on the other collar in a groove cut parallel to the axis of the beam. This allowed compensation for the upward and downward movement of the points of support of the deflectometer which took place as the beam deflected. The deflectometer may be seen on the test specimen in Fig. 4.

To obtain the corrections necessary to be applied to the deflectometer readings to compensate for the unavoidable errors caused by vibrations, change in temperature, or other uncontrollable or accidental movement of the gage, standard readings of the deflectometer were taken on the top flange of a 24-inch steel I-beam which was not under load.

Ames-gage micrometers were used to measure the slip of the ends of the bars in beam 5L1. The reinforcement in this beam consisted of two 1¼-inch plain round bars placed one in the top of the beam and the other one in the bottom, as shown in Fig. 5. Holes were drilled and tapped in the ends of these bars and short pieces of steel were screwed in place. The gages were clamped

to these extension rods with the plungers bearings against the ends of the beam. Gage No. 1 was mounted at the east end of the top bar, gage No. 2 at the east end of the bottom bar, gage No. 3 at the west end of the top bar, and gage No. 4 at the west end of the bottom bar.

For each unit the number of cycles of load was shown by a revolution counter connected to the main shaft of the unit.

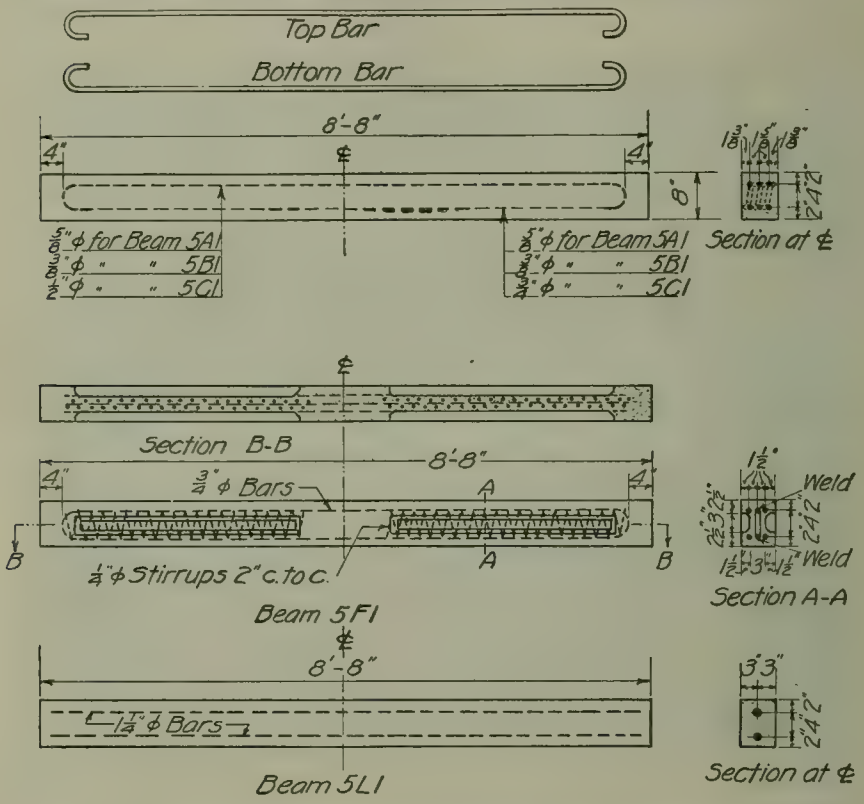

FIG. 5.—*Dimensions and reinforcement of test specimens*

III. TEST SPECIMENS AND TESTING

1. DESCRIPTION OF TEST BEAMS

The beams were 8 feet 8 inches long, 6 inches wide and 8 inches deep, and the test span was 8 feet. The top bars were placed generally 2 inches below the top surface, and the bottom bars 2 inches above the bottom surface. The cross section was rectangular for all of the beams except 5F1. That beam was I-shaped in cross section, both flanges being 6 inches wide and 2½ inches deep; the web was 3 inches thick, and the total depth of the beam was 8 inches. At the ends, where beam 5F1 was supported, and

between the load points the web was filled out to the full 6-inch width of the flange in order to avoid serious secondary stresses due to the concentration of load at these points. Beam 5F1 was made with an I-shaped cross section in order to develop a high shearing stress in the web without affecting appreciably the resistance to tensile and compressive stresses.

The reinforcing bars in all of the beams except 5L1 had semicircular hooks on the ends to furnish anchorage. Beam 5L1 was reinforced with straight bars in order to study the effect of repetition of high bond stresses on the end slip of unanchored bars.

The depth of embedment of the reinforcing bars was designed to be 2 inches in all cases. In general, it was desired to develop high compressive stresses in the concrete, and this could be accomplished in these tests only by placing the bars farther from the compression surface of the beam than otherwise would have been done. Fig. 5 shows the arrangement of the reinforcement in the beams.

Table 1 shows the amount of reinforcement used, the loads applied, and the computed stresses for all of the test specimens.

TABLE 1.—Schedule of Beams for Reversal-of-Stress Tests

Beam No.	Reinforcing bars				Load applied	
	Top		Bottom			
	No.	Diameter	No.	Diameter	Upward	Downward
		Inches		Inches	Pounds	Pounds
5A1	3	⅝	3	⅝	5000	5000
5B1	1	⅜	3	⅜	2380	2380
5C1	3	½	3	¾	670	3630
5F1[a]	3	¾	3	¾	5000	5000
5L1	1	1¼	1	1¼	6000	6000

Beam No.	Computed stresses						Remarks
	Tension in steel		Compression in steel		Compression in concrete		
	Top	Bottom	Top	Bottom	Top	Bottom	
	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²	
5A1	23 590	23 590	4293	4293	2020	2020	
5B1	28 800	28 800	−112	−112	1538	1538	
5C1	4 800	12 050	480	5080	311	1360	
5F1	16 750	16 750	5360	5360	1641	1641	I section; computed shear 175 pounds per square inch.
5L1	19 100	19 100	5900	5900	1835	1885	Computed bond stress 161 pounds per square inch.

[a] Web reinforced with one-fourth-inch wire loops, 2-inch pitch.

2. MATERIALS

A Portland cement was used which passed the tests prescribed in the United States Government Specification for Portland Cement [3] and which appeared to be of good quality in all respects.

Potomac River washed sand and gravel were used. The sand was graded in size, and was all less than one-fourth inch. The gravel was graded in size from one-fourth to one-half inch. All of the aggregate appeared to be clean, and sections sawed from some of the beams showed a good gradation of the materials, as shown in Figs. 7 and 8.

The reinforcing steel used in the test beams was purchased in the open market. Coupons for testing were taken from the steel before the beams were made. After the reversal of stress tests had been completed additional specimens of steel were taken from the broken beams and were subjected to chemical analysis and tests of their physical properties. The results of the physical tests on the individual test specimens are given in Tables 2 to 5, and the chemical analyses are given in Table 6. Eighteen specimens of the steel taken from the tested beams passed the cold bend test by being bent through an angle of 180° without any sign of fracture.

TABLE 2.—Physical Properties of Reinforcing Steel of Beam 5A1

SAMPLES TAKEN BEFORE MAKING THE BEAM

Sample No.	Location in beam	Original diameter	Yield point	Ultimate strength	Elongation in 8 inches	Reduction of area
		Inch	Lbs./in.²	Lbs./in.²	Per cent	Per cent
1		0.632	37 400	51 500	(a)	46.5
2		.629	38 500	52 700	12.5	42.0
3		.635	36 400	50 500	24.0	46.0
Average		.632	37 400	51 600	18.3	44.8

SAMPLES TAKEN FROM BEAM AFTER TEST

1	Top, north	0.637	35 950	49 600	24.8	50.4
2	Top, middle	.621	39 600	53 850	29.5	55.8
3	Top, south	.632	40 500	51 500	(a)	43.5
4	Bottom, north	.624	43 620	55 650	21.3	52.0
5	Bottom, middle	.631	38 400	52 150	25.8	43.3
6	Bottom, south	.626	39 650	53 950	20.2	57.3
Average		.629	39 620	52 780	24.3	50.4

[a] Broke outside gage length.

[3] Circular of the Bureau of Standards, No. 33; 1917.

TABLE 3.—Physical Properties of Reinforcing Steel of Beam 5B1

SAMPLES TAKEN BEFORE MAKING THE BEAM

Sample No.	Location in beam	Original diameter	Yield point	Ultimate strength	Elongation in 8 inches	Reduction of area
		Inch	Lbs./in.²	Lbs./in.²	Per cent	Per cent
1		0.381	38 830	51 890	22.0	38.0
2		.379	40 160	53 060	17.9	39.0
3		.378	44 120	50 490	13.3	16.6
Average		.379	41 040	51 810	17.7	31.2

SAMPLES TAKEN FROM BEAM AFTER TEST

1	Top, north	0.378	47 500	60 200	12.5	21.0
2	Top, middle	.377	42 350	53 950	16.4	27.5
3	Top, south	.388	40 400	52 400	18.3	34.9
4	Bottom, north	.386	39 300	55 150	18.0	25.1
5	Bottom, middle	.384	38 500	50 950	22.3	28 8
6	Bottom, south	.386	35 600	52 650	16.7	41.6
Average		.383	40 610	54 220	17.4	29.8

TABLE 4.—Physical Properties of Reinforcing Steel of Beam 5F1

SAMPLES TAKEN BEFORE MAKING THE BEAM

Sample No.	Location in beam	Original diameter	Yield point	Ultimate strength	Elongation in 8 inches	Reduction of area
		Inch	Lbs./in.²	Lbs./in.²	Per cent	Per cent
1		0.757	43 000	[illegible]	(a)	62.0
2		.746	41 100	[illegible]	28.0	55.0
3		.755	40 600	58 310	28.8	63.5
Average		.753	41 570	59 040	28.4	60.2

SAMPLES TAKEN FROM BEAM AFTER TEST

1	Top, north	0.755	41 500	60 500	28.5	65.9
2	Top, south	.754	42 500	61 300	35.5	62.9
3	Bottom, north	.750	44 100	62 000	24.0	61.5
4	Bottom, south	.755	38 600	60 700	48.0	59.4
Average		.754	41 680	61 130	34.0	62.4

[a] Broke outside gage length.

TABLE 5.—Physical Properties of Reinforcing Steel of Beam 5L1

SAMPLES TAKEN BEFORE MAKING THE BEAM

Sample No.	Location in beam	Original diameter	Yield point	Ultimate strength	Elongation in 8 inches	Reduction of [illegible]
		Inches	Lbs./in.²	Lbs./in.²	Per cent	Per cent
1		1.270	37 000	66 200	49.5	49.8
2		1.270	38 600	66 500	48.5	49.8
Average		1.270	37 800	66 350	49.0	49.8

SAMPLES TAKEN FROM BEAM AFTER TEST

1	Top	1.272	38 500	66 600	50.5	52.6
2	Bottom	1.268	39 400	66 800	49.0	50.5
Average		1.270	38 950	66 700	49.8	51.6

TABLE 6.—Comparison of Representative Physical and Chemical Tests of Reinforcing Steel Used in Concrete Ships and Reversal-of-Stress Beams

SAMPLES TAKEN FROM REVERSAL-OF-STRESS BEAMS AFTER TEST

Sample taken from—	Physical tests					Chemical contents				Grade
	Diameter	Yield point	Ultimate strength	Elongation in 8 inches	Reduction of [illegible]	Carbon	Manganese	Phosphorus	Sulphur	
	Inches	Lbs./in.²	Lbs./in.²	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	
Beam 5A1	0.629	39 620	52 780	24.3	50.4	0.023	0.20	0.130	0.064	
Beam 5B1	.383	40 610	54 220	17.4	29.8	.052	.22	.095	.052	
Beam 5F1	.754	41 680	61 130	34.0	62.4	.085	.38	.120	.095	
Beam 5L1	1.270	38 950	66 700	49.8	51.6	.31	.59	.011	.030	
Beam 5C1	.752	41 566	59 036	28.4	60.2	.085	.38	.120	.095	
Beam 5C1	.502	38 450	54 220	23.5	38.0	.....	.....	.....	.....	

SAMPLES TAKEN FROM REPRESENTATIVE TESTS OF REINFORCING STEEL USED IN CONCRETE SHIPS

Mobile	1.125	52 130	98 050	15.0	22.7	0.47	0.70	0.04	0.04	Shell discard.
Jacksonville	.625	68 420	99 130	20.5	43.0	.44	.67	.035	.035	Do.
San Francisco	1.250	37 940	60 810	24.3	.....	.21	.44	.031	.037	Structural.
San Diego	.750	36 070	61 920	25.7	.....	.22	.44	.014	.037	Do.
Wilmington	.750	45 840	63 690	25.6	54.4	.16	.38	.046	.058	Do.
Barges	[a].50	55 760	93 130	14.0	.....	.41	.50	.02	.04	Do.

[a] Square bars.

Samples of the steel taken from beams 5A1 and 5B1 for microscopic examination showed the microstructure of wrought iron which was contaminated more or less with prominent steel streaks. They had the appearance of an inferior grade of wrought iron.

The chemical tests showed that the phosphorous content in the reinforcement generally exceeded the limit for reinforcing bars,[4] and the percentages of manganese and carbon were much lower than those ordinarily found in structural steel. It seems evident that some of the steel obtained for these tests was of an inferior grade, although it was purchased as a good grade of structural reinforcement. Representative tests which were made in the inspection of reinforcing bars used in concrete ships and in concrete barge construction for the Inland Waterways Commission gave the results shown in Table 6. A comparison of the chemical analyses given in this table shows wide variations and indicates the importance of a careful inspection of all steel which is supplied for use in reinforecd concrete construction.

3. MAKING AND STORAGE OF TEST SPECIMENS

The beams for these tests were made at the Bureau of Standards, Washington, D. C. The concrete was mixed in the proportion of 1 part of Portland cement, two-thirds parts of sand, and 1⅓ parts of gravel, by weight, for all of the beams except 5C1, in which the mix was 1 part of Portland cement, three-fourths parts of sand, and 1½ parts of gravel, by weight. A rich mix was chosen in order to make the conditions closely similar in this respect to those in concrete ship construction.

The water used was 12 per cent, by weight, of the total dry materials. Calcium chloride was added to the water to accelerate the hardening of the concrete. Four pounds of calcium chloride were used with each 96 pounds of the mixing water.

A Snell mixer was used for mixing the concrete. Each batch was mixed for five minutes at a speed of 18 revolutions per minute, and the material was moved in wheelbarrows to the forms and shoveled into place. The forms used were made of wood. Beams 5A1 and 5B1 were puddled by means of one-half by 2 inch wooden paddles, while in the remaining beams the forms were rapped with a hand hammer during the period of placing the concrete. The top face of each test beam was troweled and then covered with a 6-inch board. The wet burlap, which was spread over all, was sprinkled daily until the forms were removed. The forms were removed after three days, and the beams were stored in moist sand at the normal temperature of the laboratory and kept moist during the period of storage.

[4] Standard Specifications for Billet-Steel Concrete Reinforcement Bars, A. S. T. M., Standards, p. 148; 1918.

4. CONTROL SPECIMENS

Four 6 by 12 inch cylinders, made with each beam and stored under the same conditions as the beams, were used as control specimens and were tested in compression. Results of these tests may be found in Table 7. A density calculation was made for three of the beams and is recorded in the table.

TABLE 7.—Test Results for Control Cylinders

Beam No.	Proportions of mix (by weight)	Density	Date of molding, 1918	Age of beam at start of test	Control cylinders	
					Age when tested	Average compressive strength
				Days	Days	Lbs./in.²
5A1	1:⅔:1½	0.745	Apr. 24..	153	163	5596
5B1	1:⅔:1½	.745	...do....	147	163	5563
5C1	1:¾:1½	.752	May 18..	155	139	6235
5F1	1:⅔:1½	.....	June 28..	124	98	4205
5L1	1:⅔:1½	.....	Sept. 9...	71	.....	.....

5. GAGE LINES

In beams 5A1, 5B1, 5C1, and 5F1 two horizontal gage lines, 8 inches long, were located on each side of the beam at the center. These gage lines were placed in the outside bars in both top and bottom reinforcement and are designated in Figs. 17 to 26 as north top, north bottom, south top, and south bottom.

The gage holes in the reinforcing bars were generally drilled with a No. 50 drill (0.07 inch) to a depth of about $\frac{3}{32}$ inch. They were countersunk slightly with a $\frac{3}{32}$-inch drill in all cases except for beam 5B1, the only beam which had bars as small as three-eighths inch in diameter.

In beam 5L1, which had only one bar in the top and one in the bottom, the gage lines were located only on the south side at the center of the span.

6. METHOD OF TESTING

The surfaces of the beams were whitewashed in order to facilitate the detection of cracks in the concrete during the tests. After a beam had been properly placed in the testing machine, a complete set of initial readings was taken as a basis for determination of deflections and deformations. In the case of beam 5L1 initial readings were also taken of the four Ames-gage micrometers which

were attached to the ends of the reinforcing bars for measuring the slip.

After the initial readings had been taken, the predetermined total load was applied statically in the downward direction and a new set of readings of deformation, deflection, and crack widths was taken. The cracks were traced on the beam with a pencil. The same operation was repeated with the upward load applied statically and the downward load released.

The machine was then started running and the test proceeded. In the conduct of the tests it was not possible to keep the machine running all of the time, but, when circumstances permitted, the machine was operated continuously day and night. For beams 5A1, 5F1, and 5L1 the continuity of the tests is shown in Fig. 25. For beam 5B1 the continuity is shown in Fig. 13. The vertical lines in these graphs indicate periods of rest.

At periodic intervals two readings of the strain gage were taken on each gage line, first with the upward load on the beam and then with the downward load. It was not possible to take these readings for upward and for downward load on consecutive reversals of load, but they were taken as close together as possible. Strain-gage readings were also taken at intervals with the machine not running and with no load on the test beam. When beam 5L1 was tested the slip readings for the upward load and then for the downward load were taken while the machine was in operation and as nearly simultaneously as possible.

Deflection readings were taken at intervals when the machine was in operation for the load upward and then for the load downward and generally preceding each series of steel deformation readings. Deflection readings were also taken with the machine not running and with no load on the test beam. Readings were taken on the reference beam before and after each set of deflectometer readings.

Crack widths were measured by estimation to the nearest 0.001 inch by means of a steel scale graduated to 0.01 inch and read through a glass magnifying about 4½ diameters. The crack widths were observed with the machine stopped and with the full static load on the beam. This was done for both the upward and the downward loads. Careful observations of the beam were made during the progress of the test and the cycle number at which each crack appeared was recorded on the beam just opposite the crack. As the crack increased in length the number of cycles

was recorded for various stages of the crack. Figs. 9 and 11 are views taken after completing some of the tests and show the progress of the cracks for the beams which were tested to failure.

The cracks were all outlined by means of a heavy pencil in order to make them visible in the photographs. The test of beam 5C1 was discontinued before failure occurred, and the cracks which had developed up to the time of stopping the test are sketched in Fig. 10. Each value of crack width plotted in Figs. 12 to 16 is one-half of the sum of all of the crack widths on both sides of the beam, as measured about one-fourth inch below the top or above the bottom of the beam. Each value represents approximately the total extension either in the upper or the lower surface of the beam due to the opening of cracks.

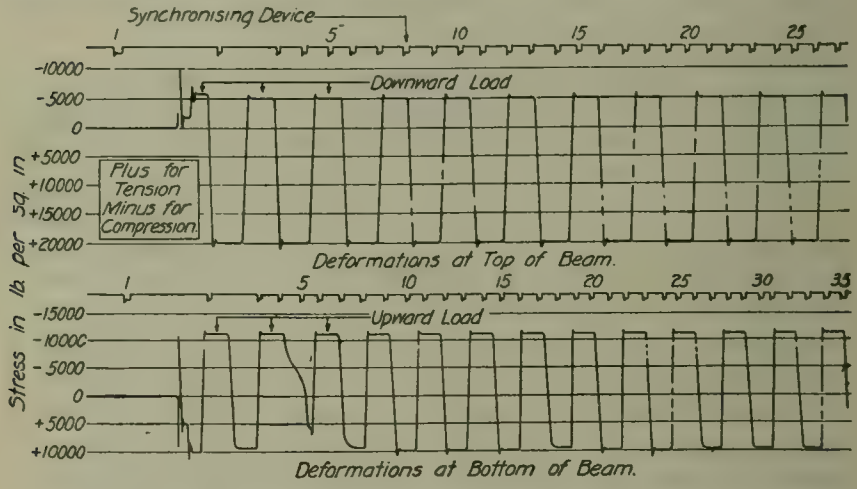

FIG. 6.—*Strainagraph record showing characteristic behavior of beams during test*

A strainagraph record,[5] taken during a portion of the test of one of the specimens, is shown in Fig. 6. This brings out some of the characteristics of the action of the testing machine.

It is found that the full load was in position about 80 per cent of the time and that about 10 per cent of the time of each cycle was required for the application of the load and 10 per cent for its removal. Assuming 17 cycles per minute as the ordinary rate of operation, it is found that the application of each load required about three-fourths of a second, and that for each 24 hours in which the machine operated the specimen was under full load about 20 hours.

[5] F. R. McMillan, The Strainagraph and its Application to Concrete Ships, Proc. Amer. Concrete Inst., 15, p. 108; 1919.

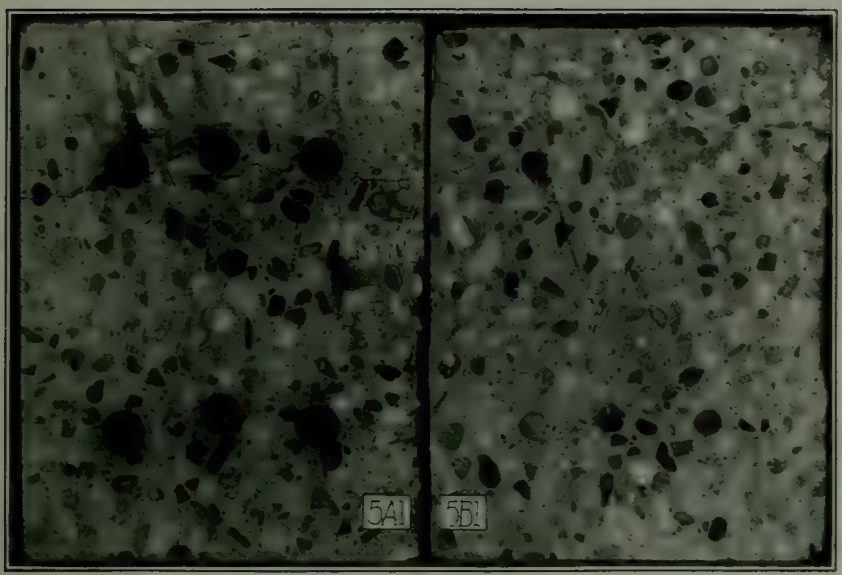

FIG. 7.—*View of cross sections through beams 5A1 and 5B1 after test*

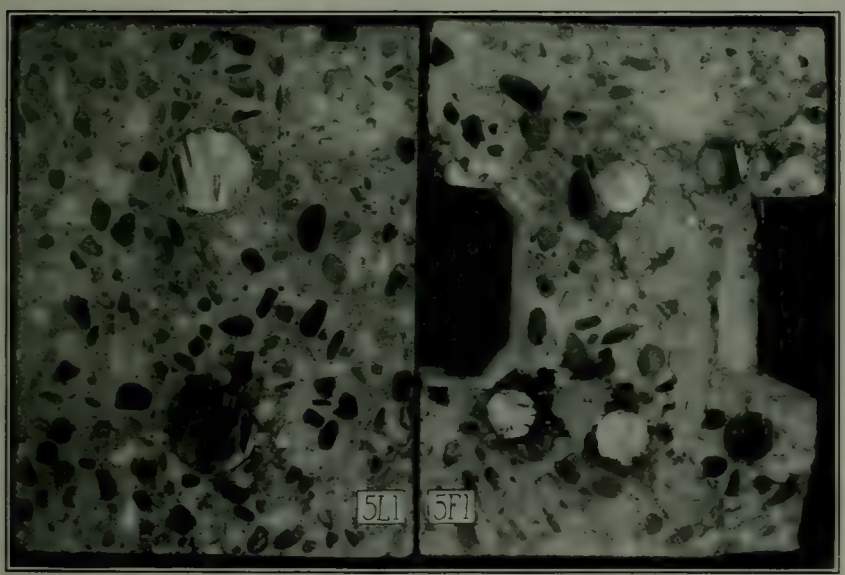

FIG. 8.—*View of cross sections through beams 5L1 and 5F1 after test*

FIG. 9.—*Side view of beams 5A1 and 5B1 after test*

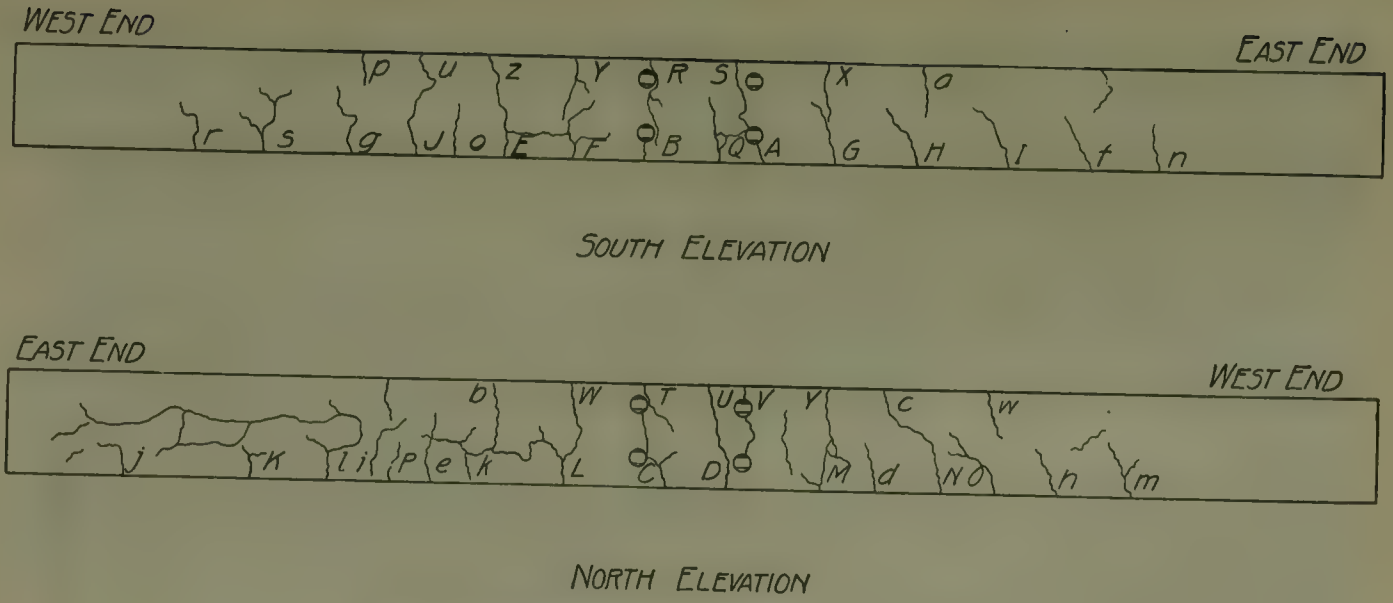
WEST END
EAST END
p u z Y R S X o
r s g J o E F B Q A G H I t n
SOUTH ELEVATION
EAST END
WEST END
b W T U V Y c w
r j K li p e k L C D M d NO n m
NORTH ELEVATION

FIG. 10.—*Sketch showing location of cracks on beam 5C1 after test*

That the stress due to impact was small is shown by the form of the graph. The only portion of the graph in which anything like an effect of impact appears is the peak which occurs with each application of the load. The height of this peak above the horizontal portion of the graph is about 4 per cent of the total height of the graph. Assuming that the peak is caused entirely by impact on the beam, the stress due to impact would be about 4 per cent of the sum of the tensile and compressive stresses due to static load. Since the inertia of the mechanism of the strainagraph may have been partly responsible for the occurrence of the peak, it is possible that the impact may have been smaller than the amount stated, but it does not seem that it can have been larger than that.

IV. TEST DATA AND DISCUSSION

1. TABLES AND DIAGRAMS

The measured dimensions and computed properties of the test beams are given in Table 8.

TABLE 8.—Measured Dimensions and Computed Properties of the Test Beams

Dimension	Notation	Beam numbers				
		5A1	5B1	5C1	5F1	5L1
Nominal cross section	.....	6 x 8 in.	6 x 8 in.	6 x 8 in.	I section	6 x 8 in.
Width of beam, inches	b	6.03	6.00	6.00	.....	6.00
Distance, compression face to center of tension steel, inches	d	6.00	5.93	6.00	6.00	6.00
Distance compression face to center of compression steel, inches	d′	2.05	2.07	2.00	2.00	2.00
Sectional area bottom steel, square inches	A_s	.9204	.3312	1.325	1.325	1.227
Sectional area top steel, square inches	A'_s	.9204	.3312	.588	1.325	1.227
Ratio A_s/bd	p	.0254	.0093	.0368	.....	.0341
Ratio A'_s/bd	p′	.0254	.0093	.0163	.....	.0341
Ratio of depth of neutral axis to distance compression face to center of tension steel	k	.461	.348	[a].530 [b].393	.495	.490
Ratio moment arm of resisting couple to depth d	j	.808	.884	[a].794 [b].823	.....	.784
Distance between center of compression area and center of tension steel, inches	jd	4.848	5.242	[a]4.764 [b]4.938	4.76	4.704

[a] Downward. [b] Upward.

NOTE.—In these computations n is taken as 10.

FIG. 11.—*Side view of beams 5F1 and 5L1 after test*

General results for the beams tested are given in Table 9. The values of the measured tension and measured compression in the reinforcement given in this table are determined directly from the unit deformations given in Figs. 17 to 23. These values for each beam represent the stresses at a time when the beam had reached approximately a stationary condition of stress. The compressive stresses given were based on the assumption that the total compressive stress in the concrete was equal to the numerical difference between the total tension and the total compression in the reinforcement of the beam. The stresses were assumed to be dis-

TABLE 9.—Results of Reversal-of-Stress Tests

Beam No.	Reinforcement					Measured tension in steel f'_s		Measured compression in steel f'_s	
	Top		Bottom		Bar ends	Top, load upward	Bottom, load downward	Top, load downward	Bottom, load upward
	Number of bars	Diameter	Number of bars	Diameter					
		Ins.		Ins.		Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²
5A1	3	5/8	3	5/8	Hooked	21 600	19 500	5400	7000
5B1	3	3/8	3	3/8	do	21 900	22 800	.....	.....
5C1	3	1/2	3	3/4	do	1 500	11 000	7500	.....
5F1[a]	3	3/4	3	3/4	do	11 400	11 700	6900	7000
5L1	1	1 1/4	1	1 1/4	Straight	18 000	17 400	4000	4100

Beam No.	Computed compression in concrete f_c		Range of stress in steel from tension to compression		Number of cycles at failure	Remarks
	Top, load downward	Bottom, load upward	Top	Bottom		
	Lbs./in.²	Lbs./in.²	Lbs./in.²	Lbs./in.²		
5A1	1565	1555	27 000	26 500	709 041	Three top bars failed in tension at east gage point.
5B1	1210	1160	21 900	22 800	59 377	Three top bars and one bottom bar failed in tension at east gage point.
5C1	157	1435	9 000	11 000	.....	Test discontinued at 2 008 000 cycles without sign of failure.
5F1	.....	.....	18 300	18 700	544 448	Top middle bar, all bottom bars, and the web steel failed in tension.
5L1	2080	2020	22 000	22 200	431 821	Both bars failed in tension 16 inches west of center.

[a] Web reinforced with one-quarter inch wire loops, 2-inch pitch: I section.

tributed according to a straight-line relation. The equation which gives these stresses is

$$f_c = \frac{2(f_s A_s - f'_s A_c)}{b(xy + d') - A_c}, \text{ in which}$$

f_c = extreme fiber compressive stress in concrete,
f_s = observed tensile stress in reinforcement,
f'_s = observed compressive stress in reinforcement,
A_s = area of reinforcement in tension,
A_c = area of reinforcement in compression,
b = breadth of beam,
y = depth from center of compression reinforcement to center of tension reinforcement,
d' = distance from compression surface to center of compression reinforcement,
$x = \frac{f'_s}{f'_s + f_s}$ = ratio of distance between neutral axis and compression reinforcement to distance between tension and compression reinforcement.

The summations of the crack widths which were measured at intervals during the tests, at levels one-fourth inch above the bottom and one-fourth inch below the top of the beams, are shown in Figs. 12 to 16. These figures show also the measured deflections. Letters were used in alphabetical order to identify the cracks as soon as they were discovered. These letters may be distinguished in Figs. 9 to 11. Figs. 17 to 23 show the number of cycles plotted as abscissas and the unit deformations as ordinates.

2. BEHAVIOR OF BEAMS DURING TESTS

In the following paragraphs are given the more important phenomena of the tests.

(*a*) Beam 5A1 had three ⅝-inch plain round bars in the top and three in the bottom. The applied load was 5000 pounds both upward and downward. This beam was tested to show the effect of repeating a compressive stress in the concrete of about 2000 pounds per square inch, combined with a relatively large range of stress from tension to compression in the steel. A stress of 1500 pounds per square inch was used in the design of the concrete ships.

Fig. 12 shows the relation between the deflection either upward or downward and the total crack widths in the top and in the bottom of the beam throughout the test. Figs. 17 and 18 show

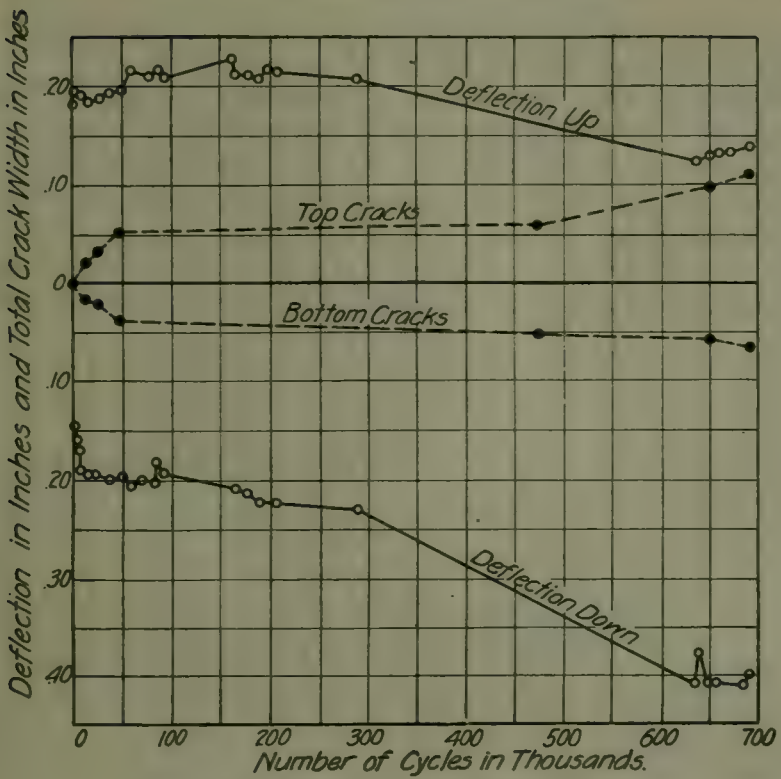

FIG. 12.—*Deflection and total crack widths for beam 5A1*

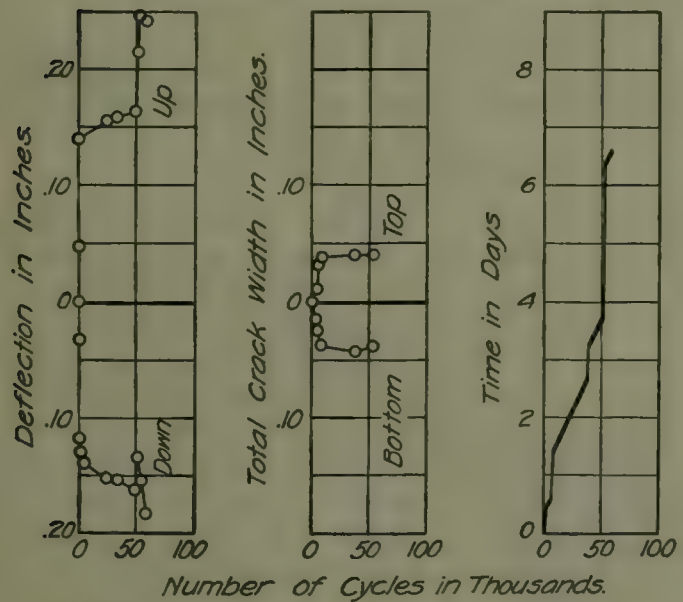

FIG. 13.—*Deflection crack widths and continuity of test for beam 5B1*

the unit deformations in the reinforcing steel as ordinates and the number of cycles of the machine as abscissas.

During the first 1000 cycles[6] the widths of the tension cracks increased to a total of 0.025 inch in the top and 0.020 inch in the bottom of the beam (Fig. 12). As the tension cracks increased in number and size the steel deformations changed slightly with a marked increase in deflection of the beam both upward and downward. After the first 1000 cycles the crack widths, deflections, and deformations remained almost constant through about

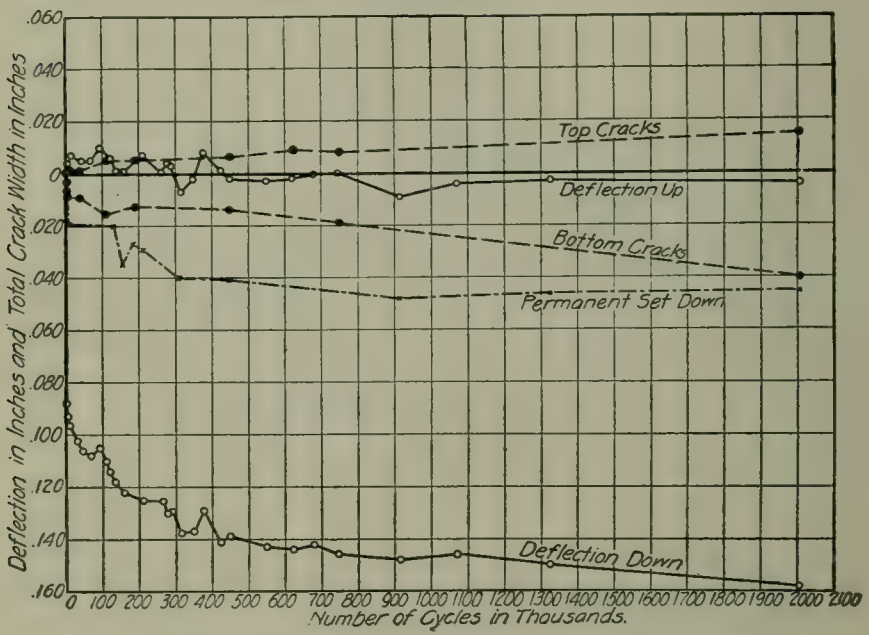

FIG. 14.—*Deflection and crack widths for beam 5C1*

one-half the duration of the test, when the deflection downward and the crack widths increased gradually until the beam failed.

The cracks increased in length, and toward the end of the test many of them extended from the top of the beam to the bottom. The cracks in the top of the beam did not close entirely with downward load, due, apparently, to some small particles of concrete chipping off and falling into the cracks. As a result the cracks in the top of the beam appeared somewhat ragged, but in the bottom of the beam they were still clean cut. The top steel failed in tension at 709 041 cycles at the west gage point, which was 4 inches west of the center of the span.

[6] An error has been found in Fig. 12. For all plotted points in this figure showing crack widths the number of cycles should be reduced by 13 500.

(*b*) Beam 5B1 had three ⅜-inch plain round bars in the top and three in the bottom. This beam was designed so that when the load was applied the computed tensile stress in the steel would be high (28 800 pounds per square inch), and the computed

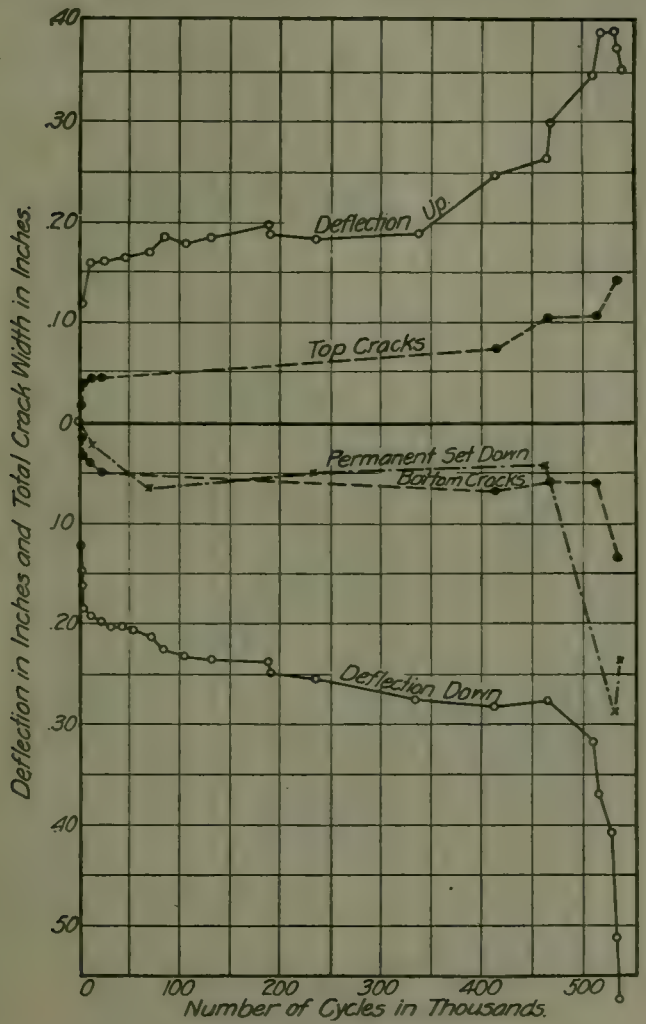

FIG. 15.—*Deflection and crack widths for beam 5F1*

compressive stress in the concrete would be about equal to the working stress used in the design of the concrete ships. The applied load was 2380 pounds both upward and downward. The deflections, crack widths, and the continuity of the test are shown in Fig. 13. The deformations are shown in Fig. 19.

No cracks appeared when a static load of 2380 pounds was first applied, but cracks *A* to *P*, Fig. 9, occurred on the first repetition of the load. During the first 300 cycles the deflections increased rapidly, while the crack widths increased rapidly during the first 7000 cycles. Both deflections and crack widths then remained

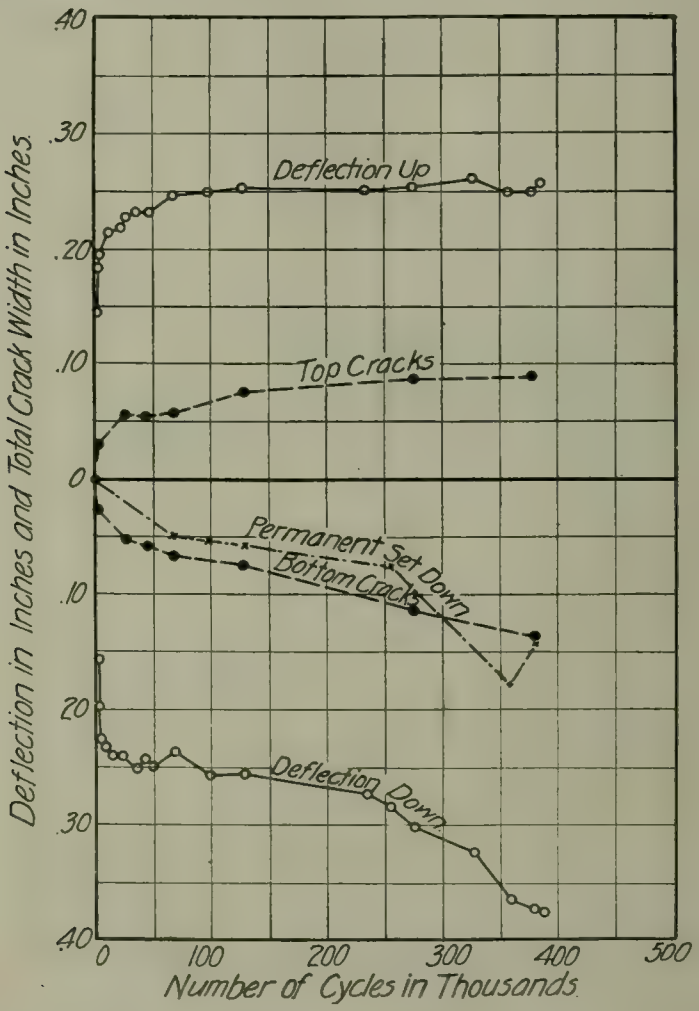

FIG. 16.—*Deflection and crack widths for beam 5L1*

almost stationary until the beam was close to failure. The deformations show less regularity than the deflections and crack widths throughout this portion of the test. In the last measurements taken before failure the deflections and deformations generally showed a great increase, but the crack widths did not.

At 59 377 cycles all three of the top bars broke without any necking which could be detected by visual observation. The south bar broke in the east gage hole, the north bar in the west gage hole, and the middle bar at a point opposite the west gage hole.

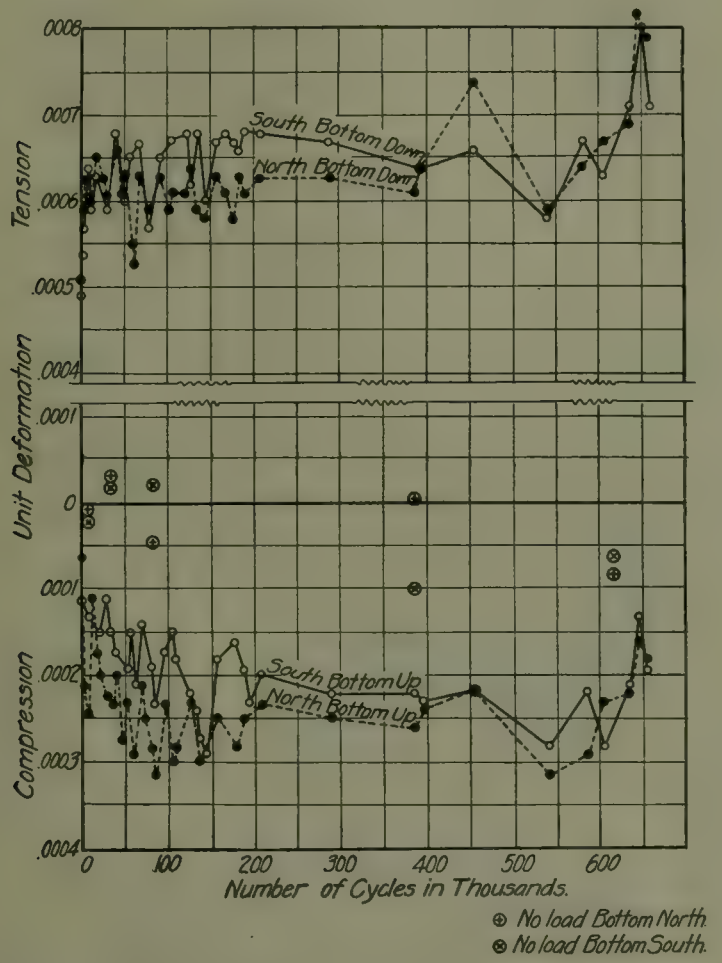

FIG. 17.—*Unit deformations in bottom of beam 5A1*

(*c*) Beam 5C1 had three ¾-inch plain round bars in the bottom of the beam and three ½-inch plain round bars in the top. This beam was designed so that the test load would produce stresses not greater either in tension or compression than the working stresses used in the concrete ship design. The applied load was 3630 pounds in the downward direction and 670 pounds in the

upward direction. Fig. 14 shows the crack widths and deflections throughout the test. Fig. 20 shows the unit deformations as the test progressed.

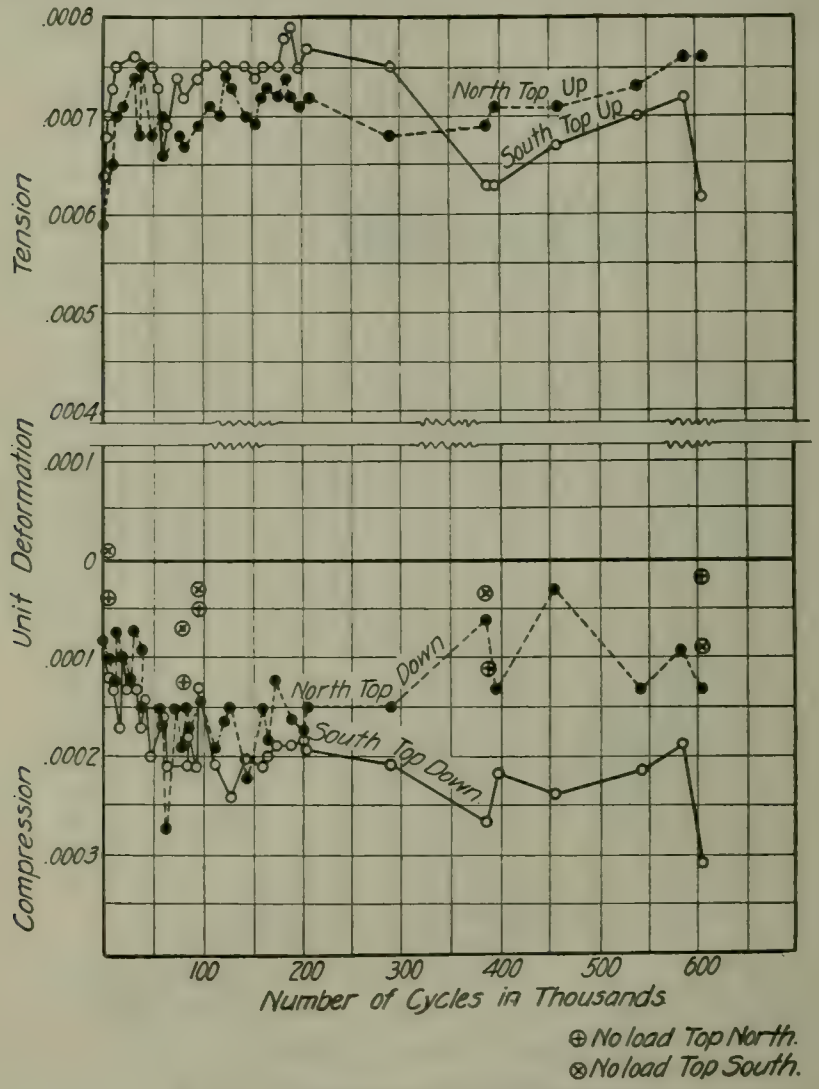

FIG. 18.—*Unit deformations in top of beam 5A1*

The upward deflections and the crack widths in the top of the beam increased slightly at the start but remained almost constant throughout the remainder of the test. The downward deflection, the downward permanent set, and the crack widths at the bottom of the beam increased gradually through about 400 000 cycles

of load. The test was discontinued at 2 008 000 cycles of load and had shown very little change after passing 400 000 cycles. The cracks were very small and extended only a short distance from the top and bottom surfaces of the beam. No serious

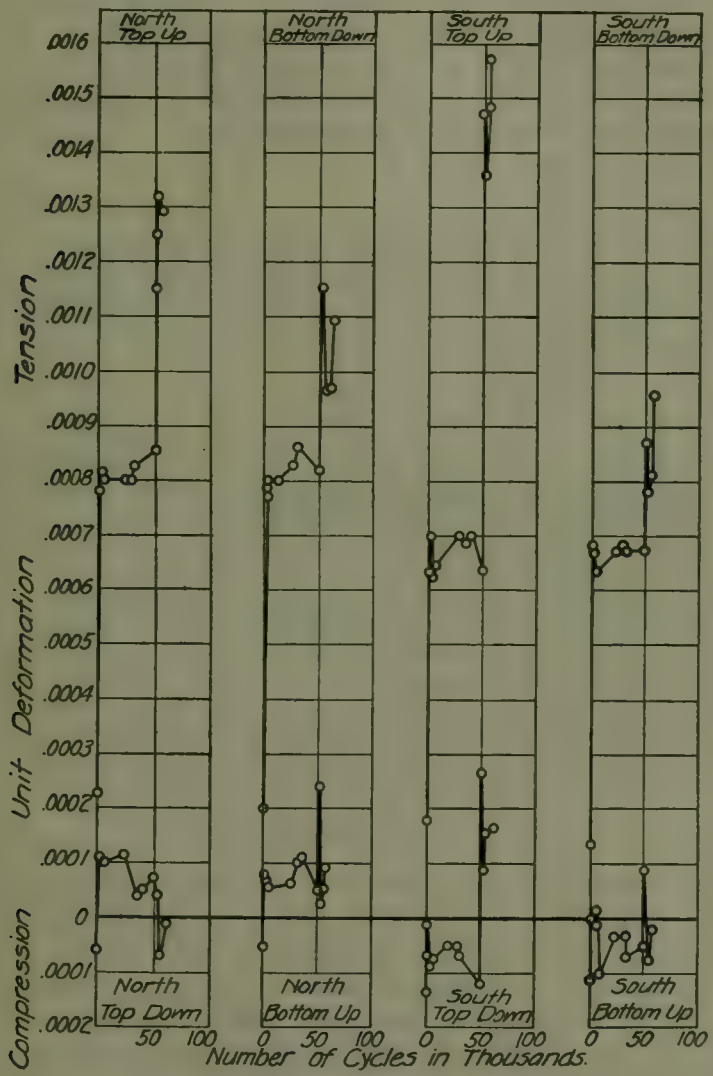

FIG. 19.—*Unit deformations in beam 5B1*

developments of any kind showed indications of approaching failure.

(*d*) Beam 5F1 was made with an I-shaped cross section with three ¾-inch plain round bars in the top flange and three in the

bottom flange, with web reinforcement consisting of a ¼-inch plain round bar wound around and welded to the top and bottom central bars with a pitch of 2 inches. This test was designed to develop high shearing stresses in the web without affecting appreciably the resistance to tensile and compressive stresses. The applied load was 5000 pounds both upward and downward. This gave a computed shearing stress of 175 pounds per square inch. Fig. 15 shows the deflections, crack widths, and permanent set downward as the test progressed. Figs. 21 and 22 show the unit deformations.

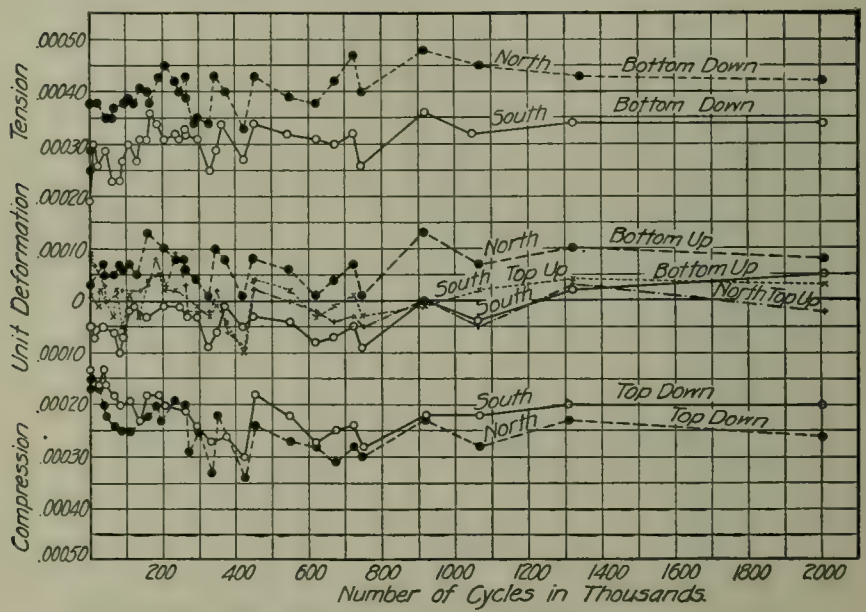

FIG. 20.—*Unit deformations in beam 5C1*

The deflections in both directions of loading increased rapidly with a gradual increase in crack widths during the early stages of the test. At 3600 cycles a horizontal crack appeared at the top of the web extending the whole length of the I-shaped portion east of the center of the beam. From the horizontal crack small diagonal cracks extended downward toward the center of the web as the test continued. Similar cracks appeared later at the junction of the web with the lower flange. From 3600 cycles until the test was about three-fourths completed, the crack widths and deflections remained almost constant but the unit deformations varied considerably.

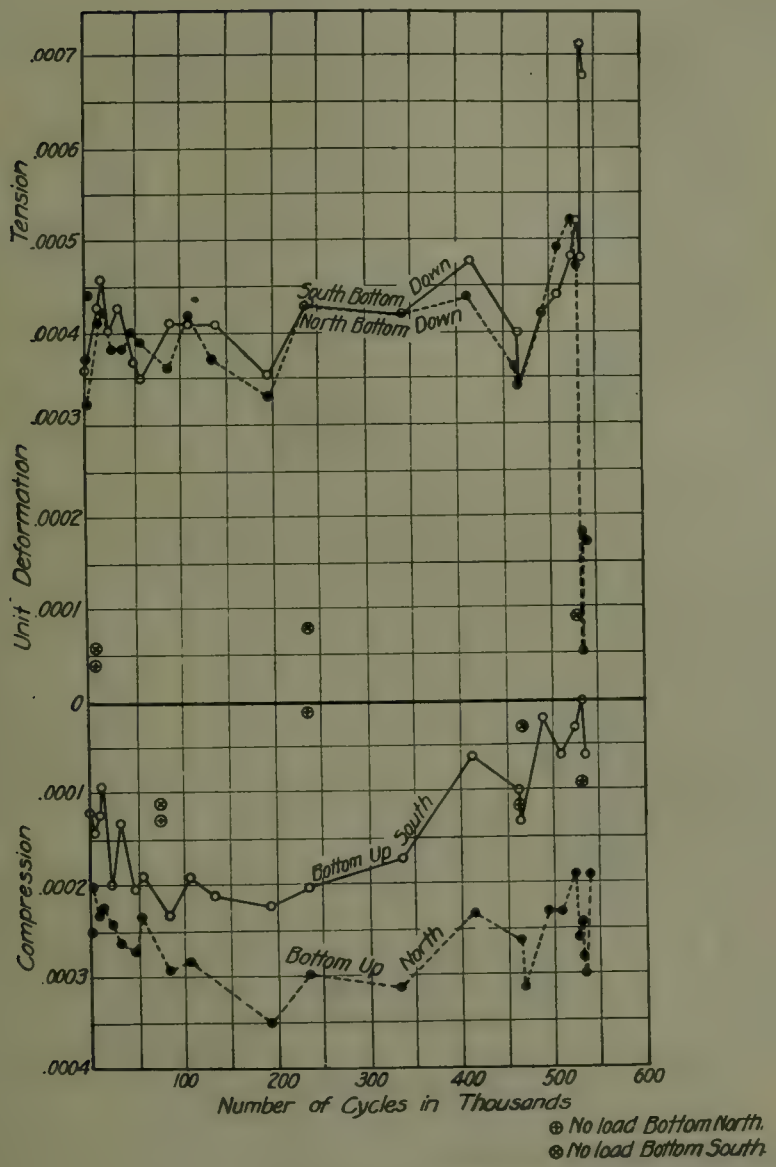

FIG. 21.—*Unit deformations in bottom of beam 5F1*

At 467 000 cycles the cracks in the top and bottom flanges and the vertical web cracks just east of the east load point, at the section where the web thickness was reduced, showed a marked increase in size and failure seemed imminent at this point. Rust and pulverized concrete were working out of the cracks at this

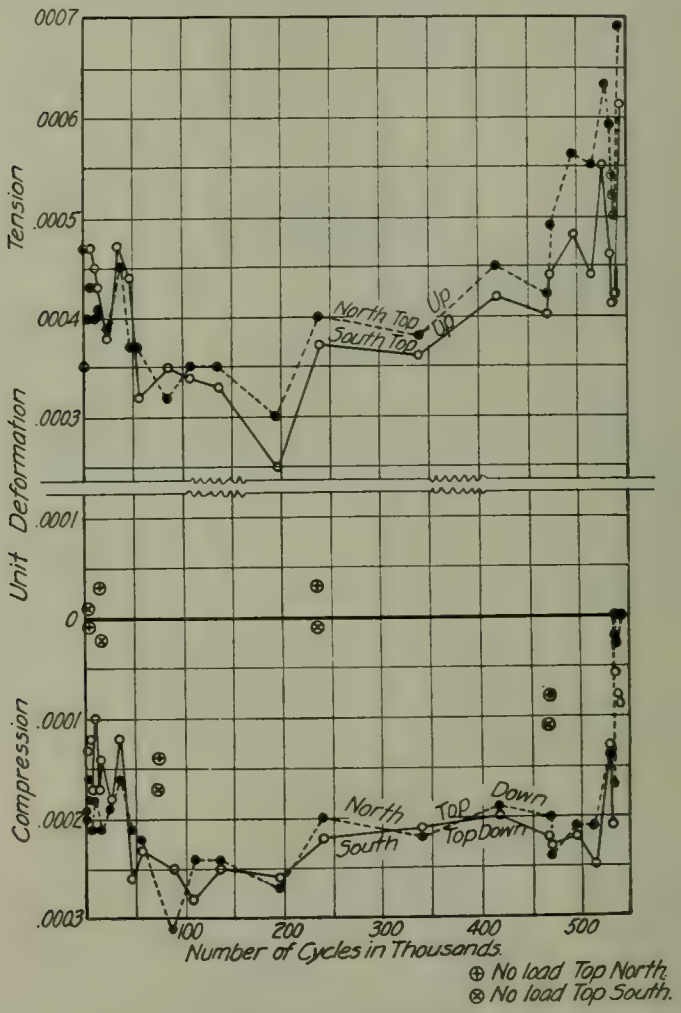

FIG. 22.—*Unit deformations in top of beam 5F1*

point and along the horizontal crack under the top flange of the beam. Apparently the horizontal crack passed under the horizontal bars and separated the top flange from the web in the east portion of the beam. A horizontal movement of the flange longitudinally with reference to the web was observed. This

movement was 0.015 inch when first measured. As the separation of flange and web became more nearly complete the movement increased, until shortly before failure it was as much as 0.07 inch.

The total crack width just before failure was 0.141 inch at the top surface and 0.133 inch at the bottom surface of the beam. The steel bars became visible in several places where the concrete was chipping off and considerable slipping of the bars in the top was noticeable with the sliding of the top flange. There was little cracking of the concrete in the west portion of the beam.

During the last 100 000 cycles of the test the deflections and permanent set downward increased very rapidly, with an increase in the deformations in the reinforcing steel. At 544 000 cycles the top and bottom middle bars and both outside bars in the bottom failed in tension at points about 5 inches east of a section through the east gage holes. The web reinforcement in that section also failed in tension at the point where it was welded to the top bar. Although the end of the test was brought about by complete tension failure in the reinforcement, the separation of the flanges from the web was so extensive that, toward the end of the test, the beam sagged greatly and some of the load probably was carried by the lever which was not applying the load.

(*e*) Beam 5L1 had one 1¼-inch plain, round, straight bar in the top and another in the bottom. The beam was designed for the purpose of studying the effect on the end slip caused by repeated application of high bond stresses. The applied load was 6000 pounds both upward and downward. This gave a computed bond stress of 161 pounds per square inch. The permanent set downward, the crack widths and the deflections are shown in Fig. 16. The deformations in the steel are shown in Fig. 23. Fig. 24 shows the end slip of the bars.

A gradual increase in deflections, crack widths, and permanent set downward took place until 70 000 cycles were recorded. The top crack widths and upward deflection remained almost constant until the completion of the test. The permanent set downward, the downward deflection, and the bottom crack widths increased gradually throughout the remainder of the test.

Up to 7200 cycles very gradual movements of the ends of the bars from their initial positions were detected by the gages which were used to measure slip, but no reversal in direction of movement with the reversal of load could be detected. From this time on

throughout the test there was a rapid increase in the amount of slip at the west end of the bottom bar and a reversal in the direction of the movement of the end of the bar with the reversal in the direction of load. It was expected that failure would occur on account of this slip, but it actually occurred after 431 821 cycles of load by tension in both reinforcing bars at a section 16 inches from the center of the span.

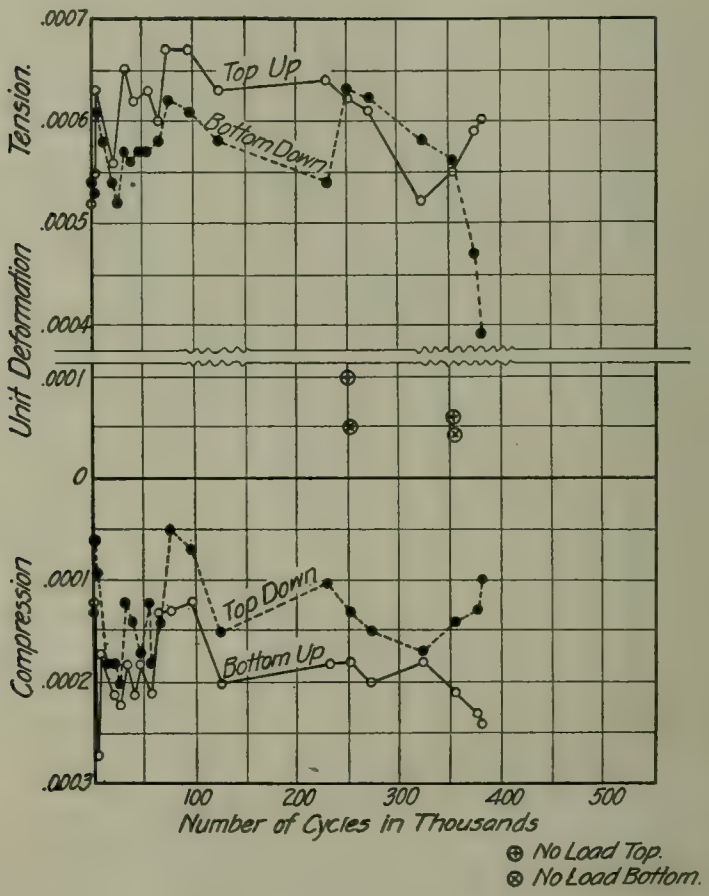

FIG. 23.—*Unit deformations in beam 5L1*

3. SIGNIFICANCE OF TENSION FAILURES

The failures of all of the beams tested have been by tension in the steel. The failures generally occurred with fewer repetitions of load than would be expected if the results of previous tests [7]

[7] Moore and Seely, The Failure of Materials under Repeated Stress, Proc. Am. Soc. Test. Mats., **15**, p. 437, 1915; also Constants and Diagrams for Repeated Stress Calculations, **16**, p. 471, 1916.

be taken as a guide. It seems probable that the use of defective steel as reinforcement was partly responsible for this result. Yet it should be noted that the steel was purchased as a commercial grade of reinforcement and probably was no worse than steel that is frequently used on small jobs where little or no inspection is exercised. All of the tension failures of the steel occurred without necking of the bars. This is characteristic of failure under repeated tensile stress and, whatever may have been the grade of the steel used, the effect of the repetition of loads seems clearly to have been present.

Besides the tension failures in the steel, other effects of the loading were apparent, and if a better grade of steel had been used, it seems probable that in some cases failure would have been due to other causes than tension in the reinforcement. Thus, in beam 5A1 a ragged appearance at the edges of the cracks was noted, and if the steel had not failed when it did, critical conditions in the concrete might have developed. However, the indications at the time of failure were that the concrete was still carrying its full proportion of the compression.

In beam 5F1 the web was badly cracked before the tension failure occurred. The flanges of the beam had been almost detached from the web at one end and the deflections had become excessive. It is possible that the tension failure may have been hastened by a repeated bending of one of the reinforcing bars. At any rate, it seems likely that a failure would have developed in the web before a great length of time, if failure of the steel in tension had not intervened.

In beam 5L1 the end slip of one of the bars had increased to 0.06 inch before tension failure took place. This is well beyond the amount of slip which, in the analysis of test data,[8] has previously been recognized as critical, and it seems certain that a complete failure in bond was impending and would have been the primary cause of failure if the tension failure in the steel could have been delayed for a short time.

4. END SLIP OF BARS

Slip of bars was measured in beam 5L1 only. In all other beams the bars were hooked to prevent end slip. Fig. 24 shows the movement of the ends of the bars plotted as ordinates, and the number of cycles of load as abscissas. Movement toward the

[8] D. A. Abrams, Tests of bond Between Concrete and Steel, Bull. No. 71, Eng. Exp. Sta., Univ., of Ill., 1913.

center of the span is plotted upward and movement away from the center is plotted downward.

Most of the slip occurred at the west end of the bottom bar. Up to 7200 cycles of load a movement of 0.0009 inch at this point

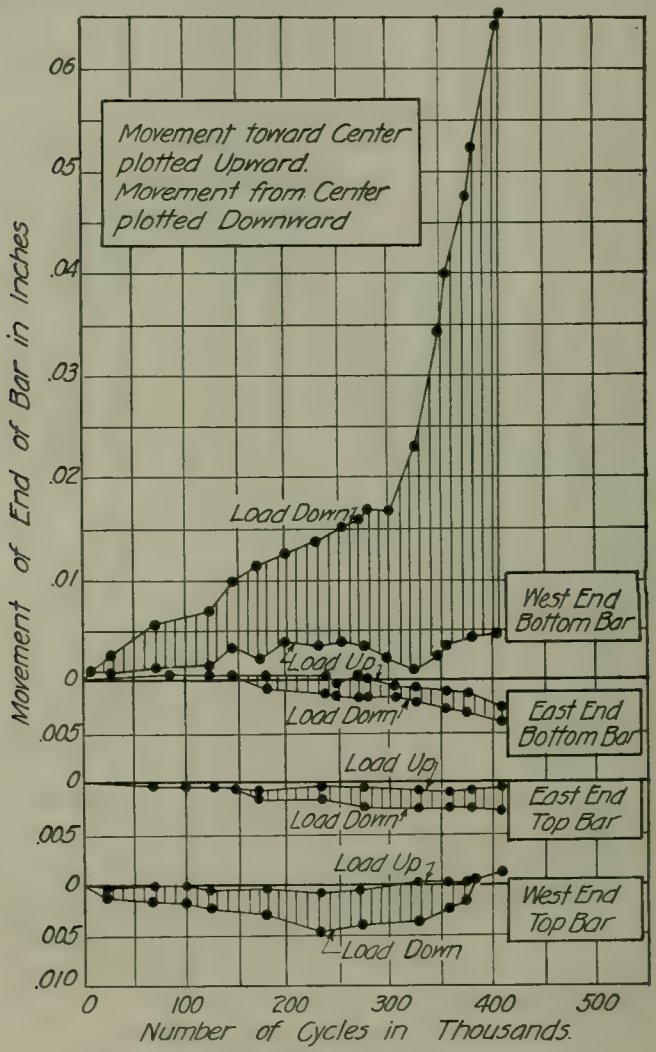

FIG. 24.—*End slip of reinforcing bars for beam 5L1*

was detected. This may have represented an actual movement of the bar with reference to its initial position, but up to this stage of the test no movement of the pointer of the Ames dial could be detected between successive applications of the load. From this time on, however, there was an instantaneous movement of the west end

of the bar toward the center of the span when the load was applied downward, and a return to approximately the original position when the load was applied upward. This cycle of slip was traversed with each cycle of load, and as the bar reached the end of its slip the suddenness of its stopping was marked by a sharp sound as of the impact of steel against concrete. The movement toward the center of the span increased with an increase in the number of applications of load, but as the test progressed the end of the bar fell short more and more of coming back to its original position with the application of upward load.

It is a matter of interest to note that the data indicate a simultaneous movement of one end of the bottom bar toward the center of the span and of the other end away from it; that is, the entire bar would appear to have been moving through the beam from west to east. The total movement of the bottom bar was slight, however, and it is doubtful if the data afford any satisfactory explanation of this phenomenon. It is just as inexplicable that both ends of the top bar appear to have been moving away from the center of the beam as that the bottom bar, as a whole, was moving through the beam from west to east. However, the movements observed at the ends of the top bar were so small that they are unimportant in comparison with the large amount of slip which occurred at the west end of the bottom bar.

The sharp increase of slip which occurred at the west end of the bottom bar at 300 000 cycles of load seemed to forecast that failure would be due to the slipping at that position; but, after 431 821 cycles of load, failure occurred by tension in both of the reinforcing bars at a section 16 inches west of the center of the span. When the beam was broken up an examination showed that the adhesion between the lower bar and the concrete in the west portion of the beam had been entirely destroyed. Near the section of tension failure scratches on the reinforcing bar were present, which indicated an abrading action of the aggregate on the bar, due to its movement back and forth through the concrete.

It is of importance that, although up to 7200 cycles of load there was no measurable slip between successive applications of load, a continuation of the test finally produced a slip of 0.06 inch. The slip probably would have progressed to the point of causing failure of the beam if tension failure in the reinforcement had not occurred before that stage was reached.

5. EFFECT OF GAGE HOLES ON LOCATION OF POINT OF FAILURE

As noted in Part III, article 5, the gage holes in the reinforcing bars were 0.07 inch in diameter and about three thirty-seconds inch deep. In most cases also the holes were countersunk slightly. The proportional reduction of area of the cross section of the bar due to the presence of the gage holes was comparatively small, even in the case of the smallest bars used, which were three-eighths inch round, but the effect of the gage holes in hastening failure may have been important. It has been pointed out [9] that "a sudden change of section is a great source of weakness in withstanding repeated stresses." The presence of gage holes furnished a sudden though slight change in section.

The smallest bars used would be expected to show the largest effect from the presence of gage holes. In beams 5A1 and 5B1, having five-eighths and three-eighths inch bars, respectively, failure occurred through the gage holes. In these beams the middle bars had no gage holes, but in each case failure of the middle bar occurred at a point just opposite a gage hole in the outer bar.

In all of the beams there seemed to have been a tendency for failure to occur in all of the bars at the same section through the beam. For the beams having three-eighths and five-eighths inch bars, the location of the section of failure appeared to be influenced by the location of gage holes. For the beams having three-fourths and 1¼ inch bars, the location of the section of failure was not determined by the location of the gage holes.

The number of repetitions required to cause failure was smallest for the beam 5B1, in which the proportional effect of the gage holes was largest. This seems to be in keeping with the discussion in the previous paragraphs, but the tensile stresses developed in this beam, as shown by the deformations in Fig. 19, were enough larger than those in the other beams to raise some doubt as to the correctness of the conclusion that the gage holes had an important effect in hastening failure or determining where it should occur.

If the gage holes in the five-eighths-inch bars were important in causing failure, beam 5A1 should have been expected to fail under a smaller number of applications of load than beams 5L1 and 5F1, in which failure did not occur through the gage holes. On the contrary, beam 5A1 withstood a larger number of repetitions than either 5L1 or 5F1, although the stresses developed in beam 5A1 were higher than the stresses in either of the other beams.

[9] Johnson's "Materials of Construction," fifth ed., p. 776.

Although there are indications that the gage holes in the bars had some influence in determining the location of the section of failure, it does not seem that they had much effect upon the ultimate strength of the steel under repeated loading.

6. EFFECT OF CRACK DEVELOPMENT ON LOCATION OF POINT OF FAILURE

Even where gage holes in the bars appeared to determine the location of the section of failure, there was also a marked development of cracks at the same section. In fact, in all cases tension failure of the reinforcing bars occurred at sections where large cracks extended entirely through the section of the beam.

At the sections where tension failure occurred the maximum widths of the cracks just before failure were as given in Table 10. These were the largest cracks in the beams. In beams 5A1 and 5L1 it was noted that the cracks did not seem to close entirely with each reversal of load. If the fractured surfaces were abraded to such an extent as to render ineffective some of the concrete, the reinforcement would have been forced to take more than its share of the compressive stresses and that would have increased the range of stress in the steel, traversed in each cycle, above that given in computations of stress (Table 1) based upon the unity of the entire section. On the contrary, the data taken as the test progressed do not show any more rapid increase in compressive stress than in the tensile stress, and this would discredit an explanation of failure based upon an assumed increase in compression in the steel as the test progressed.

TABLE 10.—Width of Cracks at Points of Failure

Beam No.	Designation of crack measured	Measured width of crack	Number of cycles at time of measurement	Number of cycles at failure
		Inch		
5A1	B	0.012	648 241	660 000
	F	.025		
	H	.023		
5B1	A	.000	57 387	59 377
	F	.000		
	J	.013		
	N	.002		
5F1	u	.025	532 490	544 448
	F	.012		
	M	.023		
5L1	D	.070	377 450	431 821
	R	.008		
	Z	.070		
	c	.040		
	p	.040		

If the reinforcement had taken all of the compression, the stresses in tension and in compression should have been equal. The fact that the tensile stresses were much larger than the compressive stresses indicates that the concrete must have assisted greatly in resisting the compressive stresses. Although the cracks seemed not to be entirely closed, this must have been merely a surface condition.

At the sections where failure finally occurred there developed, toward the end of the tests, a change in slope of the beams so sharp that it could be observed by visual inspection without instruments. This phenomenon, whatever be its cause, would be expected to introduce a sharper curvature of the bar at this point than the elastic curvature of the beam as a whole. Bending stresses in the reinforcing bars independent of those in the beam as a unit would result, and this may assist in explaining the early failure of some of the beams.

7. EFFECT OF REPETITION OF BENDING STRESSES ON PHYSICAL PROPERTIES OF STEEL REINFORCEMENT

The physical tests indicated very little change in the properties of the steel due to the repetition of load on the beam. Table 11 shows a comparison of yield point, ultimate strength, ultimate elongation, and reduction of area for specimens of the steel taken before making the beams and for specimens taken from the beams after failure. Each value is derived from the tests of four to nine specimens. This table shows that the original values were slightly smaller than those obtained from specimens cut from three of the beams after the reversal-of-stress tests, but the original values were somewhat greater for the specimens applying to beam 5B1 than the average values obtained from specimens cut from the beam after failure. In most cases the difference is so slight that it is probably accidental.

TABLE 11.—Comparison of Properties of Reinforcing Steel after Repeated Loading with Properties before Repeated Loading

[Values are stated in percentage in corresponding values obtained before use in beam tests]

Beam No.	Yield point	Ultimate strength	Ultimate elongation	Reduction of area
	Per cent	Per cent	Per cent	Per cent
5A1	106.0	102.3	132.9	112.3
5B1	99.0	104.8	[illegible]	95.5
5F1	100.0	103.4	119.8	103.6
5L1	103.0	100.6	101.7	103.6

In the failures under repeated stress, no necking of the bars could be detected, and this is characteristic of fatigue tests of steel. Almost the same ultimate elongation was shown in the tension tests of the steel specimens taken from the bars before molding of the test beams and from the bars which had been subjected to repeated stress in the beams. This indicates that the general structure of the steel was not affected by the repeated reversal of stress.

8. CHEMICAL COMPOSITION OF STEEL REINFORCEMENT

Table 6 shows the chemical composition of the steel used as reinforcing for the test beams and also for some of the concrete ships and barges built by the Emergency Fleet Corporation. This table shows that the steel used in the test beams was generally much lower in carbon and much higher in phosphorus than that used in the ships and barges. It is generally believed that low carbon content reduces the resistance of the steel to fatigue, and that high phosphorus content reduces its resistance to shock. It has been stated that "in normal steels the strength against fatigue increases with the carbon content up to about 0.09 carbon," [10] and that "Phosphorus is especially dangerous in railroad rails which are exposed to severe shocks in service, * * *. For most purposes not more than 0.05 per cent of phosphorus is allowable in steel, except for thin rolled plates, etc." [11]

Beam 5L1 was the only one in which the chemical analysis of the steel approached that ordinarily expected in a good grade of reinforcing steel. Yet that beam failed under an unexpectedly small number of repetitions of reversed stress. This fact indicates that the poor grade of reinforcing used in the other beams is not in itself sufficient to account for the early failure of those beams.

9. EFFECT OF REST PERIODS ON NUMBER OF CYCLES THAT CAUSED FAILURE

Reference to Fig. 25 shows when rest periods occurred in the tests. There is no indication in Figs. 17 to 23 that these rest periods affected in any way the amount of the deformation. This result might be expected, but Fig. 26 seems to indicate that an increase in the proportion of the time during which the machine was not in operation caused a marked increase in the number of cycles which was required to cause failure. It is believed that the arrangement of the plotted points must be largely accidental,

[10] J. B. Johnson, Materials of Construction, fifth ed., p. 776.
[11] H. F. Moore, Materials of Engineering, p. 96.

since there were inequalities in the stresses developed which would be expected to destroy the regularity of the curves.

It is not to be expected that a given period of rest will have equal value in prolonging the life of a beam, regardless of the stage of the test at which the rest period occurs.

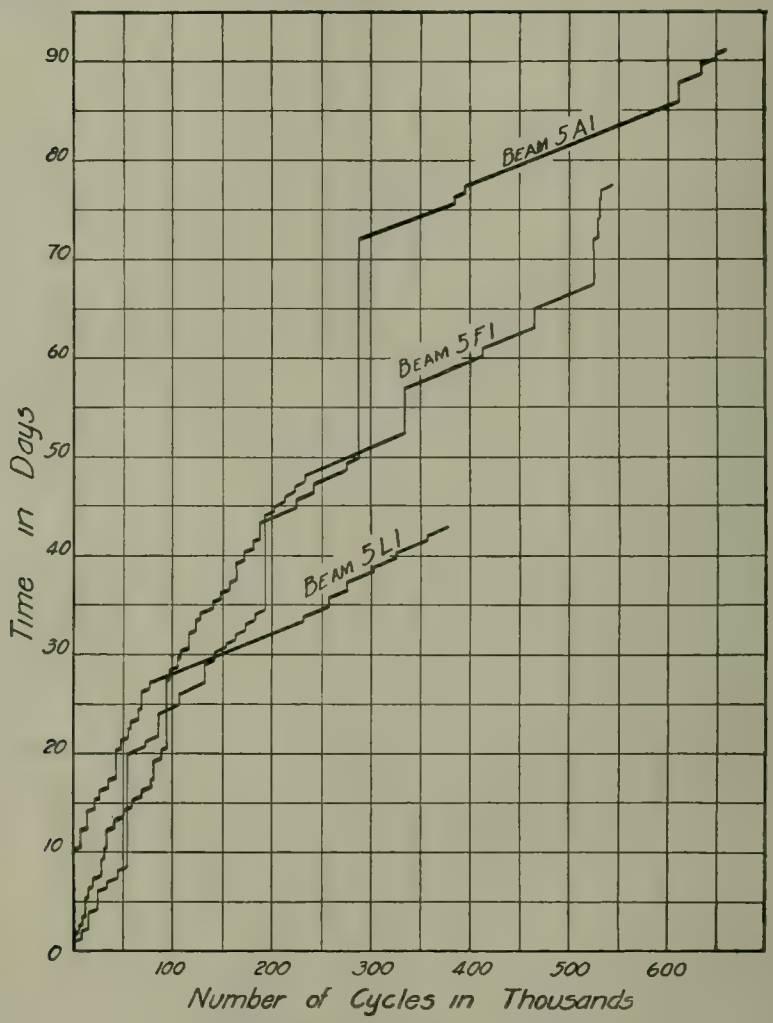

FIG. 25.—*Continuity of tests for beams 5A1, 5F1, and 5L1*

The rest periods also varied greatly in length and were irregular in their frequency.

When these facts are taken into consideration, it seems that less importance should be attached to the curves in Fig. 26 than their regularity would indicate.

10. RELATION BETWEEN MAGNITUDE OF BENDING STRESSES AND NUMBER OF CYCLES THAT CAUSED FAILURE

In Fig. 27 the abscissas represent the number of cycles of load which caused failure in all beams except 5C1 and the ordinates

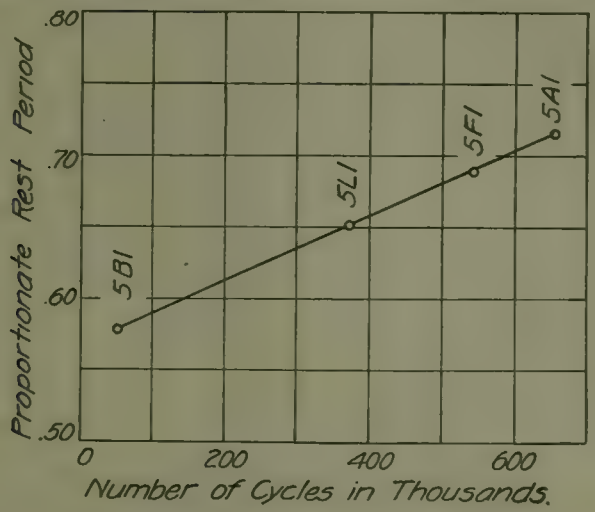

FIG. 26.—*Ratio of rest periods to total time of test*

represent the tensile stress in the reinforcing bars corresponding to the measured deformations at the same load. Fig. 28 is similar except that the ordinates represent the arithmetical sum

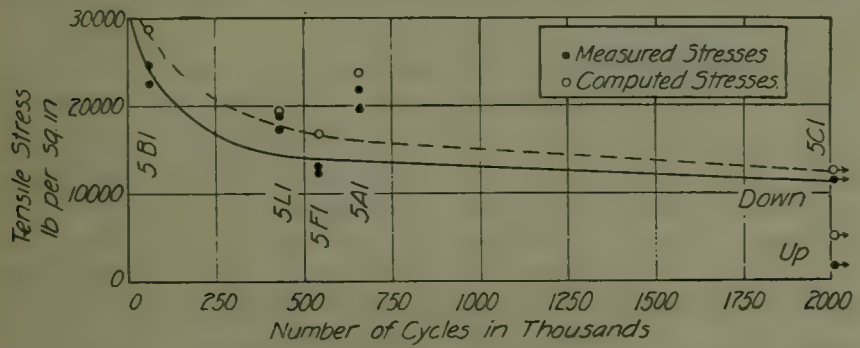

FIG. 27.—*Relation between tensile stress and number of cycles at failure*

of the tensile and compressive stresses instead of the tensile stresses alone.

The data of beam 5C1 are included in these figures although the test of that beam was not carried to the point of failure. They are used to indicate the trend of the curve although it is

recognized that the inclusion of these data is not fully justified. In Fig. 27 the stresses for beam 5C1 for the upward load are given no weight in determining the positions of the curves. The upward load was much less than the downward load, and the stresses were correspondingly smaller. Therefore it is not likely that the time of failure would depend to any extent upon the number of repetitions of the upward load.

Although the discussion in Part IV, article 7, indicates that the tension failure of the steel in these beams can not have been due directly to the large number of repetitions of the tensile stress, nevertheless Figs. 27 and 28 indicate that there is some relation between the number of cycles of load required to cause failure and the tensile stress developed. The number of cycles

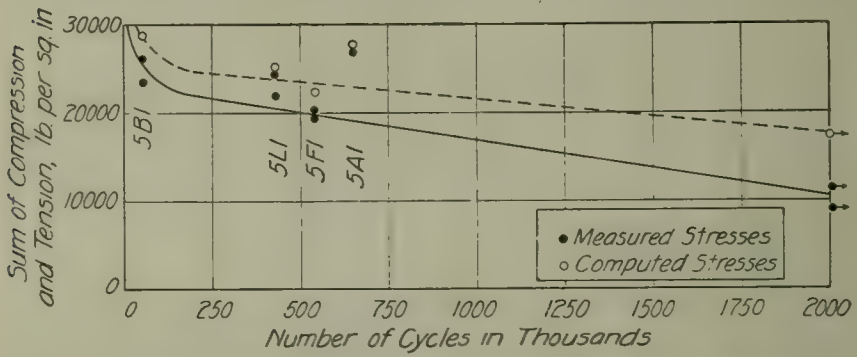

FIG. 28.—*Relation between sum of compressive and tensile stresses and number of cycles at failure*

decreased with the increase of stress, except in the case of beam 5A1.

In Part IV, article 3, it was suggested that the formation of the pronounced crack in the top flange of beam 5F1, and the consequent bending of the bar at this crack, may have been an important element in bringing about tension failure of the bar. If this explanation applied to all similar cases, bending would be likely to be more severe in beams where the tensile stresses are high than in those where they are low. This would help to account for the results shown in Figs. 27 and 28.

On the other hand, it seems that the arrangement shown in Figs. 27 and 28 may be largely accidental. In beam 5L1 large bars, 1¼ inches in diameter, were used and a slip of 0.06 inch at one end of one of the bars took place before failure occurred. Both of these facts would cause one to expect more severe stresses

at the crack due to the bending of the bars than would be expected with small bars and with no end slip of the bars. It may be this action, rather than the severity of the tensile stress plotted in Fig. 27, which accounts for the early failure of this beam.

Beam 5B1 failed under the smallest number of applications of load, and both the calculated and measured stresses were higher in this beam than in any other. However, it has been pointed out in Part IV, article 5, that the early failure of this beam may have been due as much to the reduction in area caused by the gage holes in the small bars used as to the intensity of the tensile stress.

With the possibility of explaining the failures of beams 5B1 and 5L1 on other grounds than the tensile stress produced in the steel reinforcement, and with beam 5A1 not conforming to the suggested relation between intensity of stress and number of cycles which caused failure, it seems possible that the arrangement of plotted points shown in Figs. 27 and 28 may be accidental.

11. POSITION OF NEUTRAL AXIS

The positions of the neutral axis for the beams tested are shown in Figs. 29 to 33.

Fig. 19 gives the deformations in the reinforcing bars on the south side of beam 5B1 at the top and at the bottom of the beam. A study of this figure shows that up to about 50 000 cycles of load the amount of tension was nearly the same on the top with the load acting upward as that on the bottom with the load acting downward, and that the amount of compression was nearly the same on the top with the load acting downward as that on the bottom with the load acting upward. A similar statement may be made regarding the bars on the north side, Fig. 19. These relations are as should be expected when the upward loads and the downward loads are equal and when the reinforcing bars in the top and in the bottom of the beam are at equal distances from the neutral axis. The equality of stress in top and bottom bars under conditions of tension and compression, respectively, was not preserved, in general, after the number of load cycles had exceeded 50 000.

It would also be expected that the deformations at the top and at the bottom of the beam would be the same at any instant on the north side as those on the south side, but a study of Fig. 19 shows that they were not equal for beam 5B1. The differences, however, are such as to make the range in deformation from

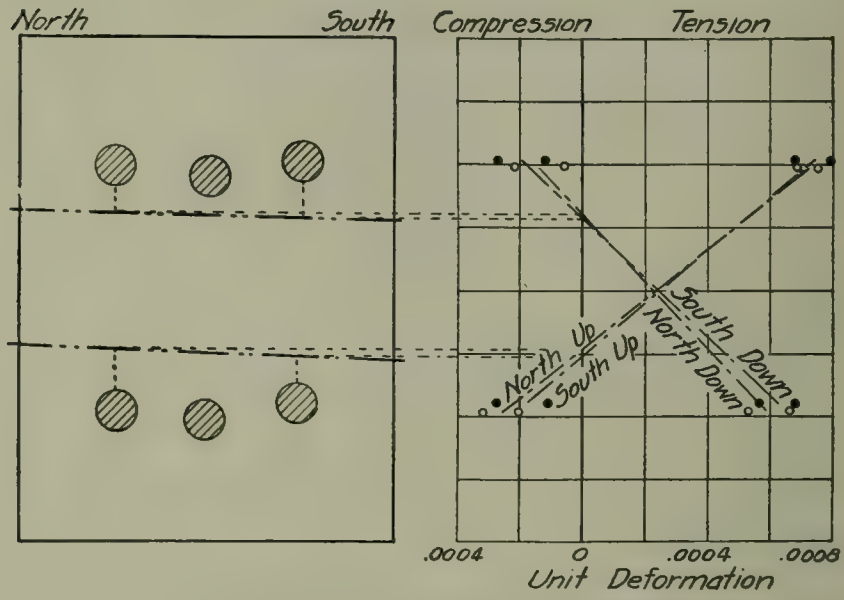

FIG. 29.—*Strain distribution and positions of neutral axis for beam 5A1*

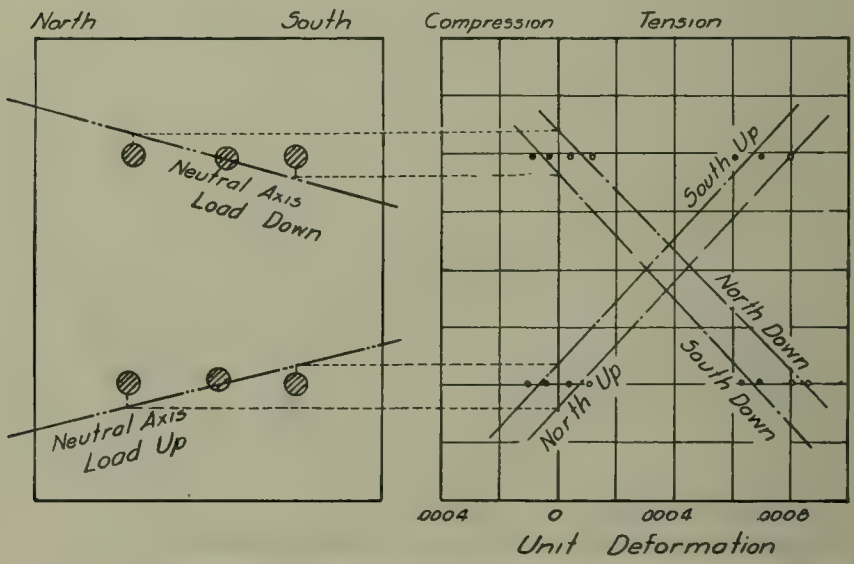

FIG. 30.—*Strain distribution and positions of neutral axis for beam 5B1*

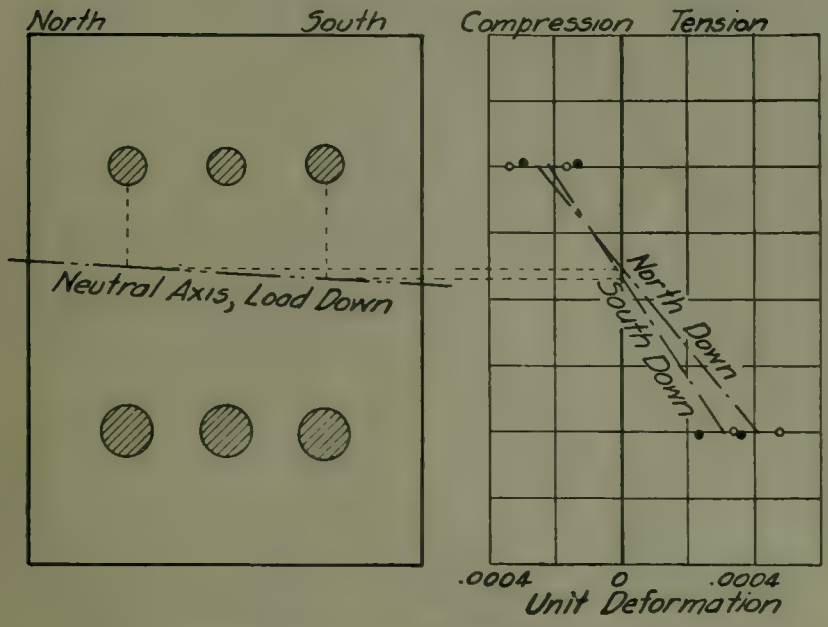

FIG. 31.—*Strain distribution and position of neutral axis for beam 5C1*

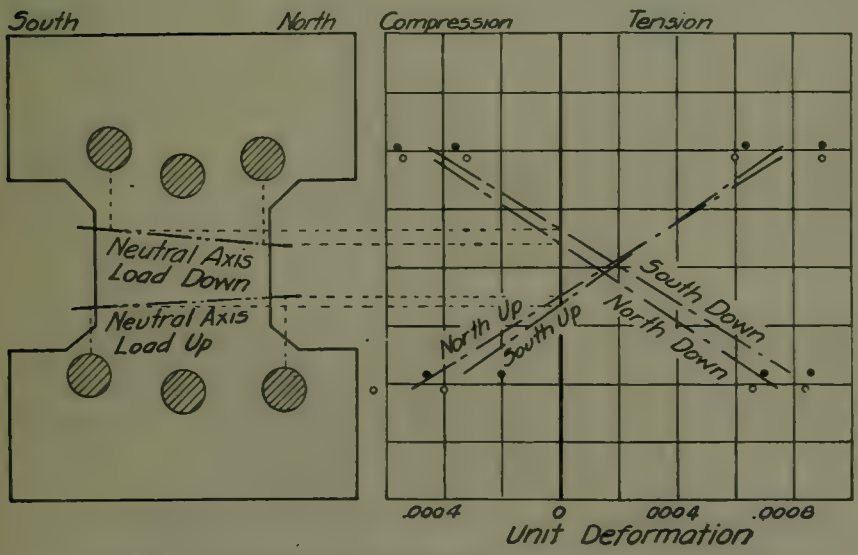

FIG. 32.—*Strain distribution and positions of neutral axis for beam 5F1*

tension to compression at any one gage line, either on the top or on the bottom, the same as the total difference in deformations between a gage line on a top bar and one on a bottom bar. These relations are made much more clear by a study of the strain distributions for beam 5B1 that are shown in Fig. 30. The individual points plotted show the extreme range of deformations corresponding to load cycles above about 10 000 and below about 50 000. The lines are drawn through the mean values represented by the points.

From these strain distributions the approximate positions of the neutral axis for the upward loads and for the downward

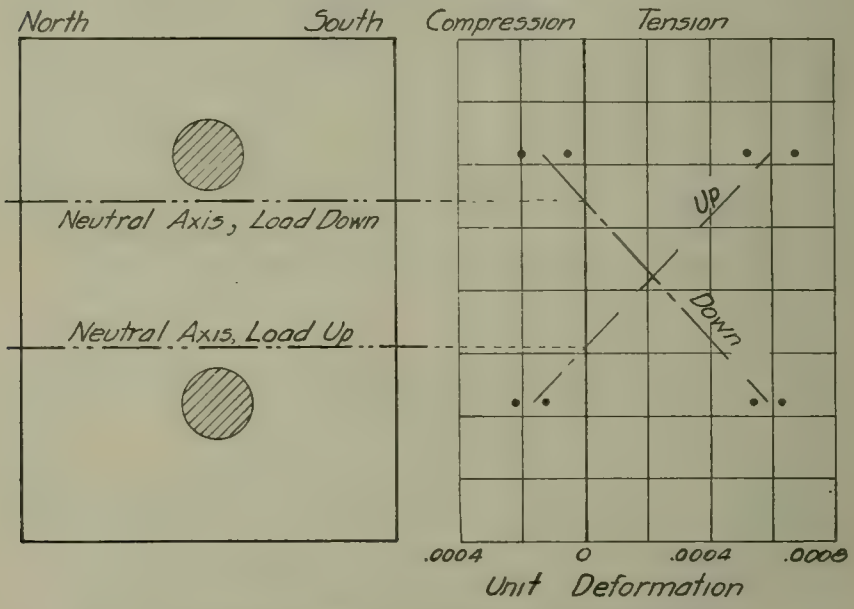

FIG. 33.—*Strain distribution and positions of neutral axis for beam 5L1*

loads have been determined and are shown in the same figure. The neutral axes under the conditions of upward and downward loading do not coincide, nor are they parallel, but the symmetry of the two neutral axes about the horizontal center line of the beam may be of some interest. The convergence of these axes could be accounted for by a curvature in the horizontal projection of the beam, but there is no evidence that any appreciable amount of warping sideways existed. However, a certain looseness in the bearings of the testing machine was observed, and it is possible that this introduced a slight inclination of the beam in one direction when the load was acting downward and in the opposite

direction when the load was acting upward. This would bring about the conditions which were observed. Another possible explanation of the convergence of the neutral axes may be found in the lack of homogeneity in the concrete, which might have considerable effect on the elasticity of opposite sides of a beam of small cross section.

Similar diagrams are shown for beams 5A1, 5C1, 5F1, and 5L1. It should be noted that the strain readings in all of these cases were taken with the machine in operation, and it is not possible to show the instantaneous position of the neutral axis because readings could not be taken on all of the gage lines simultaneously. However, the deformation curves of Figs. 17 to 23 maintain nearly enough the same positions in relation to each other to indicate that by plotting the extreme range of deformations for a small number of cycles the average would represent with reasonable accuracy the conditions which existed at a given time. This procedure has been followed in preparing the diagrams shown in Figs. 29 to 33. The deformation measurements were taken on the side of a bar at the level of its center line.

The positions shown for the reinforcing bars in Figs. 29 to 33 were determined by measurements on the sections after the beams had been tested and sawed in two, except for beam 5C1. This beam has not been sawed in two, and the positions shown are those indicated in the design of the beam.

Any error of observation is likely to show itself in an apparent inclination of the neutral axis. In Figs. 29 and 31, representing beams 5A1 and 5C1, all of the inclination present may be due to errors of observation. Figs. 30 and 32, for beams 5B1 and 5F1, show marked inclination of the neutral axis, and it is likely that these diagrams represent approximately actual conditions in the beams.

For beam 5L1 the neutral axis, as shown in Fig. 33, is horizontal. In this beam there was only one bar in the top and one in the bottom and the measurements were taken on only one side of the beam; consequently, the data furnish no basis for determining the inclination of the neutral axis, if such existed.

The data for beam 5C1 for upward load seem inconsistent and have not been plotted in Fig. 31. The peculiar showing in Fig. 20, in which compression is sometimes found where tension should be expected, is not understood. The smallness of the upward load in relation to the downward load, the consequent importance

of any residual deformations, and the importance, relatively, of small errors of observation were factors which probably contributed to the inconsistencies in the data.

12. SUMMARY

(*a*) For all of the beams tested failure was by tension in the steel. Generally the beams in which the highest stresses were developed in the test withstood a smaller number of repetitions of load than those in which the measured stresses were smaller. However, even the largest number of repetitions was so small that failure in the steel would not have been expected at the time at which it did occur considering that the observed stresses were so low. Other factors than the intensity of the tensile and compressive stresses seem to have had a part in bringing about the early tension failure.

(*b*) All of the tension failures in the reinforcing bars occurred at sections where large cracks extended entirely across the section of the beam. It is possible that in some cases the bending at these cracks was sufficient to make the bending of the bar an important factor in causing failure. The slipping of bars at the ends, such as happened in beam 5L1, would permit the opening of wide cracks and accentuate this tendency.

(*c*) The presence of the gage holes in the bars seems to have had some influence in hastening tension failure, but this influence is not very distinct.

(*d*) The quality of steel used for most of the reinforcement was poor, and this would contribute to bringing about early failure. However, this alone would not account for the small number of repetitions of stress generally required to produce failure.

(*e*) The effect of the repeated loading on the physical properties of the steel reinforcement was slight, at most; the indications of the presence of any effect probably is accidental.

(*f*) Although there appears to exist a marked effect of the rest periods on the life of the beam, it seems unreasonable to believe that the apparent effects are anything more than accidental.

(*g*) After 7000 repetitions of a bond stress of 161 pounds per square inch the slip at the end of the bar in beam 5L1 was less than 0.001 inch; that is to say, less than the amount which has been taken as the criterion of safe conditions based on tests of the bond resistance between concrete and steel. Yet, after 400,000 cycles of load, the amount of slip had increased so much that

failure by slipping of the bars seemed imminent. It seems that the intervention of tension failure at an unexpectedly small number of repetitions of load prevented the bond failure of this specimen.

(*h*) Generally the neutral axes of the beams were inclined, suggesting a slight eccentricity in the application of the load. It is not apparent that the conditions causing this inclination of the neutral axis should have anything to do with shortening the life of beams under reversal of stress.

The tests reported herein indicate the importance of conducting further research upon the effect of repeated reversals of stress in reinforced beams.

WASHINGTON, July 9, 1920.

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NOTES ON SMALL FLOW METERS FOR AIR, ESPECIALLY ORIFICE METERS

BY

EDGAR BUCKINGHAM, Physicist

Bureau of Standards

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NOTES ON SMALL FLOW METERS FOR AIR, ESPECIALLY ORIFICE METERS[1]

By Edgar Buckingham

ABSTRACT

The paper contains information, compiled for the use of physiologists of the Chemical Warfare Service, on the selection, design, and properties of small flow meters for air.

1. FORM OF ORIFICE

If an orifice meter is adopted, it is not advisable to use a very thin orifice plate, nor, if the plate is thick, to chamfer it on the outside so as to give the orifice a sharp edge. The thickness of the plate should be about one and one-half times the diameter of the orifice, and after a hole of the required diameter has been drilled through the plate it should be rounded off to a trumpet shape on the inlet side, so as to give an easy entrance. A suitable profile is a quarter circle of radius slightly less than the thickness of the plate, set tangent to the hole already drilled and to the entrance face of the plate, thus leaving the orifice cylindrical for a short distance in from the exit face.

An orifice of this sort, if carefully made and smoothly finished, will give a discharge which is within a few per cent of the so-called "theoretical" discharge. This is convenient because the size of orifice needed for a particular purpose may then be computed approximately from the theoretical equations, which is not the case for sharp-edged orifices.

[1] These notes were prepared in December, 1917, at the request of the Chemical Warfare Service and were intended primarily for the use of physiologists. Various calls for the same sort of information have made it seem desirable to publish them.

18705°—21

2. COMPUTATION OF THE THEORETICAL DISCHARGE THROUGH A FRICTIONLESS ORIFICE

Let—

D = the diameter of the orifice in millimeters.

h = the pressure drop at the orifice.

p = the pressure of the air on the inlet side, or the "initial pressure."

$p_1 = p - h$ = the pressure on the exit side, or the "back pressure."

$r = p_1 \div p$. It is assumed that the pressure drop h is not more than 0.4 p, so that the pressure ratio r is always between 0.6 and 1.0.

T = the absolute temperature of the air on the inlet side, or the "initial temperature," measured in centigrade degrees.

V = the rate of discharge of dry air in liters per minute, measured under the initial conditions p, T.

Then the theoretical discharge is given by the equation: [2]

$$V = 2.12\, D^2 \sqrt{T} \sqrt{r^{\frac{10}{7}} - r^{\frac{12}{7}}} \qquad (1)$$

It may be noted that the absolute values of the pressures do not appear explicitly in this equation. If the initial and final pressures are both doubled, the *mass* of air discharged per minute will also be doubled; but since V is now to be measured at this doubled initial pressure, its value in liters per minute will remain unchanged and may still be found from equation (1).

The volume which these V liters of air would occupy at any other temperature and pressure may be found by means of the familiar relation $pV = RT$. In particular, the volume V_0 which the air discharged per minute would occupy if measured in liters at 0° C and at a pressure of 760 mm of mercury, is given by the equation

$$V_0 = 0.761\, D^2 \frac{p}{\sqrt{T}} \sqrt{r^{\frac{10}{7}} - r^{\frac{12}{7}}} \qquad (2)$$

in which p is expressed in millimeters of mercury.

[2] A deduction of this equation is given in a note at the end of the paper.

3. THE RELATION OF ACTUAL TO THEORETICAL DISCHARGE

In practice, the discharge of dry air from an orifice or nozzle of the form described in section 1 is found to be a little less than the value computed from equation (1) or (2). The ratio of the actual to the theoretical discharge is known as the discharge coefficient. For orifices of a diameter of 2 mm or more the *dsicharge coefficient* will usually be over 0.9 and may be nearly 1. Hence the theoretical equation enables us to compute beforehand about what size of orifice will be needed for a given discharge under given conditions, and so to design or to select an orifice suitable for the purpose in hand.

But it is not possible to predict the value of the discharge coefficient exactly; and, furthermore, the value is not constant for a given orifice, but varies with the pressure ratio r. Hence each orifice must be standardized by experiments at various values of r. For orifices which are large enough that accurate reproduction is mechanically practicable, a single standardization may suffice for a number of similar orifices; but for diameters of only 1 or 2 mm such accuracy is difficult of attainment, and should not be assumed without investigation.

It is not possible to give exact formulas for moist air; but it may be said that, unless the air is so moist that water is deposited in the orifice, the discharge will not differ much from that for dry air. If water is deposited on the walls of the orifice, the discharge rate is liable to be irregular. For security, the air should be dry, when practicable, or the orifice should be slightly warmed to prevent the deposition of water upon it.

4. THE EFFECT OF VARIATIONS IN THE INITIAL TEMPERATURE OF THE AIR

While the discharge coefficient of a given orifice varies somewhat with the pressure ratio, it is not sensibly affected by moderate changes in the initial temperature of the air; and for an initial temperature range of 0 to 50° C it is safe to treat the discharge coefficient as independent of the temperature. This means that when an orifice has been standardized at one temperature, the same standardization may be used for other temperatures if the changes of temperature are allowed for as indicated in the theoretical equations already given. If the air reaching the orifice is at ordinary room temperature, in the vicinity of 20° C or 293°

absolute, a change of 1° C changes the value of V or of V_0 by a trifle more than one-eighth of 1 per cent. Hence, if no greater accuracy than 1 per cent is required, an orifice that has been standardized at 1 temperature—e. g., 18° C—may be used without any temperature correction for any temperature within 5 or 6° of the temperature at which the experimental standardization was carried out.

5. THEORETICAL DISCHARGE CURVE IN TERMS OF THE SUCTION HEAD

In using an orifice meter the quantity primarily observed is the pressure drop h; the initial temperature and either the initial or the back pressure being also observed if not already known. Having the values of h and either p or p_1 we may set $r=\frac{p-h}{p}$ or $r=\frac{p_1}{p_1+h}$ and compute the theoretical discharge by means of equation (1).

But since the computation is rather cumbersome, it is convenient, if many problems relating to the same initial conditions are to be solved, to construct once for all a curve giving V in terms of h for those conditions. Furthermore, since D enters the equation very simply, it is well to construct the curve for $D=1$ mm and allow separately for changes in D.

Let us therefore set $D=1$; and let us adopt, as our fixed initial conditions, 18° C and 1 atmosphere pressure, the air being drawn through the orifice by the suction h and the back pressure being less than atmospheric. We now have $T=291$, and equation (1) reduces to

$$V=36.1\sqrt{r^{\frac{2}{\gamma}}-r^{\frac{\gamma+1}{\gamma}}} \qquad (3)$$

Let us suppose, further, that the pressure drop h is measured in centimeters of water. Then, since a head of 760 mm of mercury is equivalent to a head of 1034 cm of water, we have to substitute in equation (3) the value $r=(1034-h)/1034$. By using various values of h we may then compute the corresponding values of the theoretical discharge V and plot a curve showing the relation of V to h for an orifice of 1 mm diameter under the given initial conditions. Such a curve is appended to this paper.

6. ILLUSTRATIONS OF THE USE OF THE THEORETICAL DISCHARGE CURVE

The method of using such a curve may be illustrated by the following examples:

(*a*) Suppose we wish to know the rate at which air under the above-described initial conditions will be sucked through a 2.6 mm orifice by a head of 27 cm of water. Reading from the curve at $h=27$ we find $V=3.08$. An orifice of 2.6 mm diameter will therefore give a theoretical discharge of $3.08\times 2.6^2=20.8$ liters per minute. Actually, the discharge will be a few per cent less than this.

(*b*) Suppose that in the foregoing example the initial temperature had been not 18 but 30° C; then the theoretical rate of discharge in liters per minute *measured at 30° C* would have been

$$20.8\sqrt{\frac{273+30}{273+18}}=21.2.$$

(*c*) Suppose that we want to select an orifice which shall discharge 85 liters per minute with a suction head of from 40 to 70 cm of water. From the curve we find that for $D=1$, at $h=40$ $V=3.72$, and at $h=70$ $V=4.84$. We must therefore use an orifice of such diameter D that $\frac{85}{D^2}$ lies between 3.72 and 4.84, which means that D must lie between 4.78 and 4.19 mm. An orifice of 4.5 mm diameter would give 85 liters per minute at the same head as would be required for an orifice at 1 mm diameter to discharge $\frac{85}{4.5^2}=4.2$ liters per minute; and we find from the curve that the required theoretical head is $h=52.2$ cm of water.

7. REMARKS

It will be sufficiently evident from the foregoing examples how the curve may be used for other temperatures than the one for which it was constructed, as well as for other diameters than 1 mm. But since the use at present proposed for the curve is merely for selecting or designing orifices which are then to be standardized by experiment, and since the discharge coefficient will always be in doubt by several per cent until this standardization has been effected, no great accuracy in using the curve is needed, and allowances for temperature changes may as well be disregarded so long as the initial temperature remains between 0 and 35° C.

To the degree of approximation now in question the curve may also be used for solving problems relating to the discharge of air against a constant back pressure of one atmosphere, the head h cm of water being now an excess pressure on the inlet side above one atmosphere. At $h = 80$, the value of V read from the curve is about 3.5 per cent greater than the theoretical discharge under these conditions if the volume is measured at 18° C and at the initial pressure of (1 atm. $+h$); and it is about 4.5 per cent less than if the volume is measured at 18° C and at 1 atmosphere. For smaller values of h these discrepancies are smaller; hence for rough purposes the curve may be used to solve problems relating to this second method of working.

If, finally, readings are desired in terms of V_0, the volume discharged measured under standard conditions, a curve may easily be constructed from equation (2). The time-consuming part of such work is the computation of $\sqrt{r^{\frac{10}{7}} - r^{\frac{12}{7}}} = B$. A table of values of B in terms of r from $r = 0.92$ to $r = 1.00$ is given at the end of the paper.

8. EFFECT OF THE SPEED OF APPROACH

The theoretical equations given above are deduced on the assumption that the initial velocity of the air, at the place where p and T are measured, is so small that its square is negligible in comparison with the square of the speed of the air through the orifice. Hence if the orifice plate is merely inserted as a diaphragm across a pipe which is not much larger in diameter than the orifice itself, the formulas as given will be considerably in error.

If the pressure drop at the orifice is small, so that the pressure ratio is not far from unity, the density of the air is not very different on the two sides of the orifice. Hence the speeds in the approach pipe and in the orifice will be approximately inversely proportional to the areas, and their squares, to the fourth powers of the diameters, so that it is not necessary to slow down the approaching air by introducing a large chamber at the place where the orifice is to be put. If, for example, the internal diameter of the housing is 5 times that of the orifice, the square of the speed through the orifice is some 625 times the square of the speed of approach, and a further increase of the diameter ratio could not have any appreciable effect on the rate of flow through the orifice.

9. REMARKS ON SOME OTHER SIMPLE FORMS OF FLOW METER

(*a*) SHARP-EDGED ORIFICES or holes in thin plates are often used in flow meters, instead of the trumpet-shaped orifices or nozzles recommended in section 1 above, and if properly standardized they are satisfactory. They have, however, the disadvantage that the discharge coefficient varies rapidly with the pressure ratio and may be as small as 0.6, so that the theoretical equations can not safely be used to give an approximate estimate of the rate of discharge by merely assuming a constant discharge coefficient of, say, 0.95, as may be done with an orifice which has a trumpet-shaped entrance.

(*b*) THE VENTURI METER, while excellent in large sizes, could hardly be made satisfactory for rates of flow of the order of 80 liters per minute or less; and it would be difficult to construct on so small a scale as would be required unless recourse were had to the use of extremely delicate differential gages with their attendant disadvantages.

(*c*) CAPILLARY TUBE METERS in which the pressure drop to be measured is due to the resistance of a length of tube, have some defects of which the most serious is liability to obstruction by dust or condensed water vapor. For small rates of flow the tube must be either very long or very fine. Long tubes are inconveniently fragile if made of glass, while, if the tube is opaque, the presence of an obstruction can be detected only from the behavior of the tube. On the other hand, the fineness of bore needed when the tube is short, greatly increases the liability to obstruction. And though the visibility of an obstruction in a glass tube may save the observer from relying on erroneous readings of flow, it does not remove the obstruction nor obviate the necessity of either cleaning or restandardizing.

Certain other points regarding capillary tube meters may also be worth mentioning. We shall first suppose that the tube is straight, and that its length is a large multiple of its diameter (e. g., 1000) so that the resistance is only slightly influenced by the nature of the ends and is nearly proportional to the length of the tube. Such a tube may behave in either of two quite different ways.

If the flow is slow enough, the rate of discharge is directly proportional to the pressure drop—a very simple and convenient relation; but it is also inversely proportional to the viscosity of

the air in the tube; and since in the vicinity of room temperature the viscosity of air increases by about 0.3 per cent per degree C rise in temperature, no great accuracy can be achieved unless the tube is jacketed in some way, so that its temperature can be controlled and observed.

On the other hand, if the flow is very rapid, the nature of the fluid motion is entirely different; the resistance is greater and the rate of discharge increases more nearly as the 0.6 or 0.5 power of the pressure drop. And, finally, there is an ill-defined intermediate range of speeds where either of the foregoing regimes may establish itself, the nature of the motion and the relation of discharge to pressure drop sometimes changing suddenly from the first to the second. In this critical region no standardization can be relied on, and care must be taken, not only to avoid this range, but also to make sure that a standardization under one regime is not, by inadvertence, extrapolated to a point where the other regime is the actual one.

Short tubes form a transition stage between tubes which are so long that the nature of the ends is unimportant, and orifices in thin plates where only the ends remain and the middle has shrunk to nothing. We have very little information about the behavior of such tubes or even of long tubes which are not straight and cylindrical. It does not follow that such tubes may not be entirely satisfactory for use as flow meters after they have been standardized by experiment. But from the standpoint of design, they present the disadvantage that we have no simple mathematical theory which we know will represent their behavior sufficiently well to enable us to select the dimensions needed for a particular purpose with a certainty of getting approximately the desired result.

10. THE MEASUREMENT OF VERY SMALL RATES OF FLOW

For an orifice of 1 mm diameter and for the initial conditions, 18° C and 1 atmosphere pressure, we find by reading from the curve that a discharge of 1 liter per minute requires, theoretically, a suction head of about 2.8 cm of water. And since this is too small a head to be read accurately on an ordinary vertical U gage, it is evident that for rates of 1 liter per minute, or less, a more sensitive gage must be used, or, if an orifice meter is to be employed, the orifice must be less than 1 mm in diameter.

The sensitiveness of the reading of h may be increased about 10 times by means of an inclined U gage; but if much more is required, recourse must be taken to some more sensitive type of differential gage. There is no difficulty in making such gages, but they are likely to be inconvenient and sluggish; and unless both sides of the gage are equally accessible to slight accidental irregularities of pressure, such as may arise from a gusty wind or the opening and closing of doors, very sensitive gages are not easy to work with. On the other hand, orifices of much less than 1 mm diameter, even when carefully finished, are so liable to be affected by dust, dirt, or condensed water vapor that they are not to be recommended.

In view of the fact that the resistance of a tube of given diameter can be indefinitely increased by increasing the length, it seems likely that the most convenient form of meter for low rates of flow will be one in which the resistance across which the pressure drop is observed is furnished by a capillary glass tube, a long tube in coil form being preferred to a short and very fine straight tube. If the air is carefully dried and freed from dust, such a tube should be satisfactory in the sense of providing a constant resistance.

If it is not practicable or not desirable to dry the air before it reaches the meter, condensation in the tube may be prevented by keeping the tube at a temperature considerably above that of the incoming air. As already remarked in section 9(*c*), the temperature of the tube must be controlled if accurate measurements are to be made, and, if the bath in which the tube is immersed is kept well above the temperature of the incoming air, there will be no risk of condensation in the tube.

If a meter of this sort is adopted, the dimensions of the tube may be chosen so as to give a convenient pressure drop h at the desired rate of flow. Equations might be given connecting the rate of flow with the pressure drop for straight, round tubes of known dimensions, but they would not be reliable for bent or coiled tubes. In any event, such a tube must be standardized by experiment; and, if apparatus for standardization is available, it is a simple matter to select by trial a suitable tube for the purpose in hand.

It is advisable to keep the discharge rate always well below the critical range mentioned in section 9(*c*); that is, to havet he flow always so slow that the discharge rate is nearly proportional to the pressure drop *h*.

WASHINGTON, December 6, 1920.

Values of $B=\sqrt{r^{1.0}-r^{1.2}}$

h	$\frac{1034-h}{1034}=r$	$\sqrt{r^{1.0}-r^{1.2}}=B$	h	$\frac{1034-h}{1034}=r$	$\sqrt{r^{1.0}-r^{1.2}}=B$
80	0.9226	0.1424	10	0.9903	0.0523
60	.9421	.1247	7	.9932	.0438
40	.9613	.1029	4	.9961	.0332
30	.9710	.0896	2	.9981	.0235
20	.9807	.0736	1	.9990	.0166
15	.9855	.0639	0.5	.9995	.0118

NOTE

DEDUCTION OF THE "THEORETICAL EQUATION"

Let a fluid be flowing steadily along a channel from a section A, where the static pressure is p and the absolute temperature is T, to a second section A_1, where the pressure and temperature are p_1 and T_1, the sections being at the same level so that gravity may be disregarded.

Let v, K, e be, respectively, the volume, the kinetic energy, and the internal energy, of unit mass of the fluid as it passes A; and let symbols with subscripts refer to A_1. Let Q be the heat received from without by unit mass while it is between A and A_1.

By the first law of thermodynamics, the increase of energy per gram of fluid from A to A_1 is equal to the heat added plus the excess of the work done on the fluid as it enters at A over the work it does on the fluid ahead of it in issuing at A_1. Hence we have

$$(K_1+e_1)-(K+e)=Q+pv-p_1v_1,$$

Or

$$K_1-K=e-e_1+pv-p_1v_1+Q \qquad (1)$$

Let A_1 be the section of a stream issuing from a nozzle or orifice, and let A be a section farther upstream and of so much larger area that the speed at A is small, and the kinetic energy K therefore negligible. Let us also suppose that Q is negligible and the flow sensibly adiabatic. Then equation (1) takes the simpler form

$$K_1=e-e_1+pv-p_1v_1 \qquad (2)$$

This is general and applicable to any fluid.

Now let the fluid be ideal gas—that is, one for which the equations

$$\left.\begin{aligned} pv&=RT\\ C_v&=const\\ e&=TC_v+const\\ C_p&=C_v+R \end{aligned}\right\}$$

are satisfied. Within the limits of accuracy needed here, air is such a gas. By these equations we may easily reduce equation (2) to the form

$$K_1=C_p(T-T_1), \tag{4}$$

which says that the kinetic energy acquired, per gram, is equal to the product of the fall in temperature by the specific heat at constant pressure.

Let S_1 denote the arithmetical mean speed over the section A_1. If the speed were uniform all over A_1 and there were no cross currents or eddies, we should evidently have

$$K_1=\tfrac{1}{2}S_1^2 \tag{5}$$

and by (4)

$$S_1=\sqrt{2C_p(T-T_1)}. \tag{6}$$

The agreement of experiment with deductions from equation (6) is close enough to show that equation (5) is nearly fulfilled under ordinary conditions, and equation (5) is therefore adopted as an assumption, though often not mentioned as such.

Up to this point it has not been assumed that the flow was frictionless. If there is resistance due to viscosity or turbulence, the fluid will be heated by the dissipation; T_1 will be raised and S_1 diminished; but equation (6) will remain satisfied.

In short, well-formed nozzles or orifices the resistance is small, and if we neglect it altogether we may treat the expansion of the gas as not only adiabatic, but isentropic, and may then apply the familiar equations for isentropic expansion of an ideal gas, viz:

$$pv^k=\text{const};\quad T=p^{\frac{k-1}{k}}\times\text{const} \tag{7}$$

where $k=C_p/C_v$. By the second of these we have

$$\frac{T_1}{T}=\left(\frac{p_1}{p}\right)^{\frac{k-1}{k}}=r^{\frac{k-1}{k}} \tag{8}$$

So that (6) may be written in the form

$$S_1=\sqrt{2TC_p\left(1-r^{\frac{k-1}{k}}\right)} \tag{9}$$

an equation which gives the speed S_1 of the frictionless jet in terms of the initial temperature T, the specific heat at constant pressure C_p, the pressure ratio $p_1/p=r$, and the specific heat ratio k.

The volume of gas passing A_1 in unit time is $V_1=A_1S_1$; and the volume of this same mass measured under the initial conditions is

$$V=V_1\frac{v}{v_1}=A_1S_1\frac{v}{v_1}$$

or by (9)

$$V=A_1\frac{v}{v_1}\sqrt{2TC_p\left(1-r^{\frac{k-1}{k}}\right)} \tag{10}$$

Let A_1 be a circle of diameter D, and by (7) set $v/v_1=r^{\frac{1}{k}}$. Then substituting in (10) we have

$$V=\frac{\pi}{2\sqrt{2}}D^2\sqrt{TC_p\left(r^{\frac{2}{k}}-r^{\frac{k+1}{k}}\right)} \tag{11}$$

This holds for any normal units. For example: If D is in cm, T in °C, and C_p in ergs per g. per °C, V will be in cm³ per second.

For dry air at ordinary pressures and temperatures we have $k=1.40$, $C_p=1.01\times 10^7$ erg/g/°C, so that (11) takes the form

$$V\ [\mathrm{cm^3/sec.}]=3530\ BD^2[\mathrm{cm^2}]\sqrt{T°C} \tag{12}$$

where

$$B=\sqrt{r_7^{10}-r_7^{12}} \tag{13}$$

For V in liters per minute and D in millimeters, this reduces to

$$V[\mathrm{liters/min.}]=2.12\ BD^2\,[\mathrm{mm^2}]\sqrt{T°C}, \tag{14}$$

which is given as equation (1) in the body of the paper.

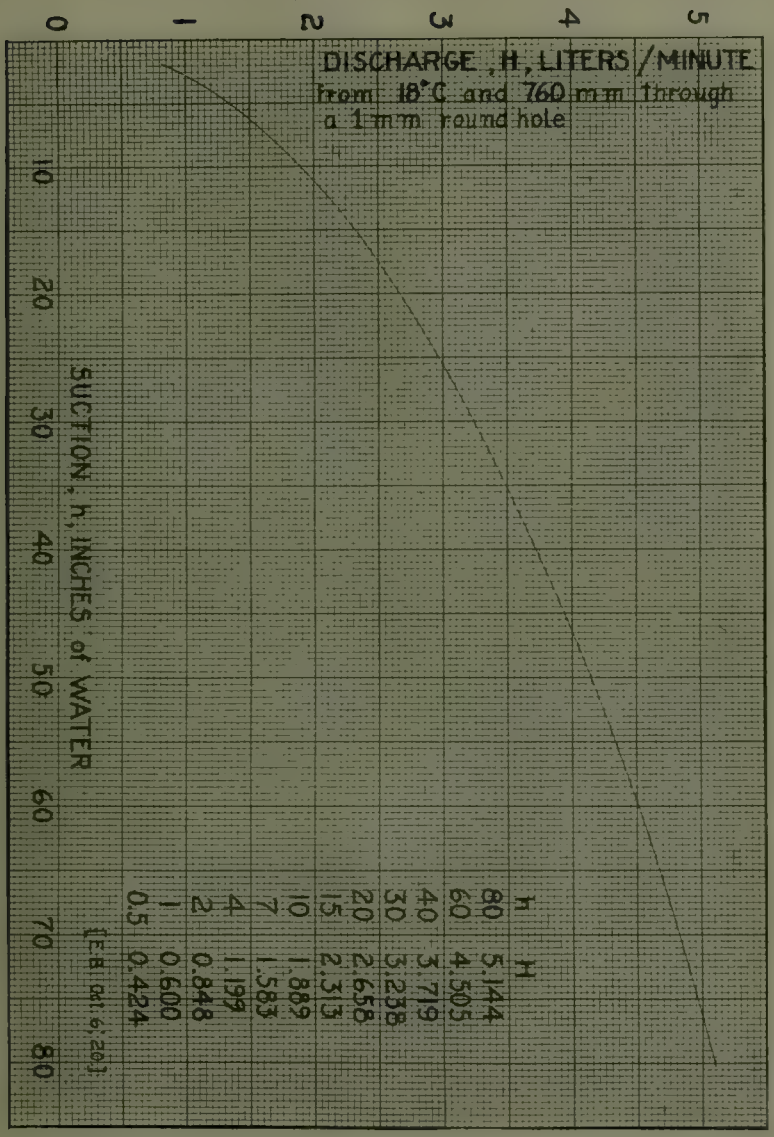

FIG. 1.—*Discharge curve*

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